NASA CONTRACTOR REPORT

NASA CR-197

NASA CR-

ELECTROFORMING ALUMINUM FOR SOLAR ENERGY CONCENTRATORS

by Ferenc J. Schmidt and Irving J. Hess

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1. SUMMARY

The aim of this program was the investigation of possible fabrication of aluminum
solar concentrators by electroforming. Since aluminum electroforming hitherto
was only a laboratory scale process, the investigation was logically sub-divided
into three major phases: (1) determination of the basic soundness of the approach
by evaluating the deposit properties from a laboratory cell; (2) scale-up of the
process and build equipment to 30" diameter concentrator size; (3) electroform and
test two 30" diameter concentrators.

In the first phase, a lithium aluminum hydride bath was chosen as the most pro-
mising electrolyte for further development. The measured deposit properties
closely approximated those of 2S aluminum: modulus of elasticity in tension =
8×10^6 psi; ultimate tensile strength = 11,000 psi; tensile yield strength at
0.2% offset = 7,800 psi; elongation = 26%; linear coefficient of thermal expan-
sion = $13.6 \times 10^{-6}/°F$; density = 2.66 g/cc; specular reflectivity of Al-SiO
vacuum-coated specimens in the 0.3 to 0.9 micron wave length range, approximately
88%; in the 1.2 to 7 micron range, approximately 95%.

In the scale-up studies, major emphasis was placed upon parameters which affect
the geometrical accuracy and surface reproduction of thin-walled electroforms.
This study necessarily included full processing sequences and the design of large
scale equipment, with particular emphasis on safety, due to the flammable and
toxic nature of the electrolyte. A number of very high quality, flat, paraboloidal
and spherical prototypes, up to 7" diameter, were prepared in this phase. It was
noted that the throwing power of the bath is significantly greater than that en-
countered with commonly-used aqueous electroforming baths, and since the ethereal
solution penetrates into deep microscopic pores, mechanical interlocking could
result within even the minutest pinholes. The relatively soft aluminum easily
distorts around such key-ins. Vigorous agitation is necessary to reduce "tree
growth". Any nodules or trees show through to the surface. Additives, to retard
tree growths, are not recommended, since they increase the stresses.

After the construction and checking out of the large scale equipment, six (6) runs
were made. Runs 1 through 4 were plagued by separation difficulties, due mainly
to interlocking of the deposit with the pinholes present in the master. In Run 5,
an initial copper flash was applied to the master to bridge across the pinholes.
This mirror separated relatively easily. In Run 5, to reduce the surface imper-
fections, the master was polished. Run 6 mirror was separated in a vacuum chamber.
While the over-all geometry of these electroforms was good, the surfaces are marred
due to three factors: (1) some local distortions were caused by interlocking
(local pull of the soft aluminum); (2) the filtration and agitation equipment were
insufficient during the runs (due to design shortcoming), and the resultant nodules
and tree-growths caused orange-peel show-throughs; (3) the vacuum coated Al-SiO re-
flective coating is somewhat fogged, due to unrelated process troubles at the
vendor's coating plant. The spincast epoxy female mold had an average slope error
of less than 1 minute. The two electroformed aluminum mirrors had average slope

errors of 6.9 minutes (copper flashed) and 9.4 minutes (all-aluminum), respectively, resulting from the factors cited above and from the mechanical polishing of the male nickel master which was necessary to minimize "keying-in" of the deposit in the pores and permit distortion-free separation of the electroform.

Despite these shortcomings, the program achieved its objectives. It was demonstrated that large scale aluminum electroforming is not only possible, but can be carried out in a safe and practical manner, and the process is suitable for the production of highly accurate, highly reflective, lightweight (approximately 0.25 lb/ sq. ft.), nonmagnetic solar concentrators. A much greater degree of perfection of substrate surface is required than in the case of nickel or copper electro- forming. The vacuum separation technique, demonstrated in this program, is a universally applicable procedure, applicable to any thin-walled, highly accurate electroform.

2. INTRODUCTION

Contract NAS 1-3309 required the development of the aluminum electroforming technology to permit fabrication of two 30" diameter paraboloidal solar concentrators.

NASA-Langley defined the general scope of work as follows:

"Explore the possibility of forming aluminum by electroforming as a possible means of constructing solar energy concentrators.

"This work should lead toward the development of the technology necessary to construct lightweight, nonmagnetic solar concentrators having a high degree of geometrical concentrating ability and highly specular reflecting surfaces. When a suitable master is used, it is desired that the method of forming will produce reflective surfaces that require no polishing or surface finishing before vacuum deposition of reflective and protective coatings."

The tasks were as follows:

1. Perform studies, both theoretical and experimental, leading to the successful forming of aluminum by electroforming.

2. Fabricate, by the electroforming method, four flat samples from masters having a surface at least equivalent to that of good quality plate glass and test these electroforms.

3. Fabricate, by electroforming, two 30" diameter paraboloidal solar concentrators.

4. Measure the geometric deviation from a true paraboloid of the concentrators and masters.

During the preceding year, GE/RSD conducted an industry-wide survey of the state-of-the-art of various aluminum precision fabricating processes, which would be potentially applicable to paraboloidal mirrors. The significant technical problems were discussed with relevant industrial establishments, research institutions and agencies. Aluminum samples* representing the best present techniques were obtained and evaluated in-house, in order to form first-hand conclusions.

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* Samples by courtesy of Battelle Memorial Institute and Reynolds Aluminum Company.

For exact replication of any surface or configuration to optical quality, the electroforming technique appears presently without equal. However, although the metal deposited on the substrate faithfully reproduces the substrate surface to better than micro-inches, inherent electrodeposition stresses tend to distort the over-all geometry. GE/RSD recently has developed a stress-controlling technique applicable to any electroforming process, which permits measurement and control of these stresses to an accuracy not previously possible.

While the literature claims of successful aluminum plating date back more than a century (Ref. 1), and by 1900 more than 20 aluminum plating patents were granted, all these claims are to be discounted. Not until Plotnikov (Ref. 2) introduced non-aqueous electrolytes was the deposit confirmed as aluminum; and only during the last decade were processes of practical significance developed, although no larger than glass beaker scale.

Brief descriptions of the three most significant modern electroforming baths are as follows:

    1. Aluminum trialkyl electrolyte yields very good, ductile deposits, but the bath is extremely poisonous (vapor, contact, etc.) and flammable.

    2. Substituted alkyl pyridinium halides yielded satisfactory baths, from which waveguides were successfully electroformed (Ref. 3). The control of this bath is difficult, and it is highly toxic and flammable.

    3. Ethereal solution containing aluminum chloride and lithium hydride, originally developed by Couch and Brenner (Ref. 4), yields very satisfactory, thick, low-stress deposits that are smooth and strong. Initial evaluation of actual samples indicated that the physical properties of the deposits should meet the requirements of a concentrator material. The deposition rate is about 1 mil/hr. The electrolyte is somewhat toxic, flammable, and explosive.

This latter, hydride type bath was used for the present program.

In the electroforming approach, the "development of technology" is largely an electrochemical process scale-up problem, with particular regard to safety considerations. It was this safety aspect of the process that largely stood in the path of its general adoption by the electroforming industry, a large segment of which is not equipped and is reluctant to handle highly flammable solutions or to operate under dry inert atmosphere conditions. There are numerous precedents to the scale-up of similar processes; some examples are; the perchloric acid electropolishing process, chlorate, perchlorate, organic per-compound electrolytic manufacture.

3. CONCLUSIONS

This program presented a considerable technological challenge, extending beyond
the usual problems of scaling up a laboratory process to practical proportions.
Electrochemical processes, as such, are notoriously difficult to scale up. Much
has been written on scale-up in other engineering fields, but very little in
electrochemical engineering, which is defined as process engineering, in which
one of the essential steps is electrochemical in nature. Electrochemical en-
gineering is one of the yougest branches of engineering, and unfortunately,
still relies heavily upon "art", if only because of the great many areas of ig-
norance existing in theoretical electrochemistry (Ref. 5).

To add to the problem, in this aluminum electrolytic process, there was only
little prior "art" to lean on. Mixed and often contradoctory results were re-
ported by the handful of investigators of the laboratory process. At least two
recent attempts at scale-up (to less than 50-gallon size) were reported as having
been abandoned for technical reasons. Design parameter, necessary for any elec-
trochemical scale up, were utterly lacking (e.g. thermodynamic properties, such
as heats of formation, free energies; activities; equilibrium constants; vapor
pressures; material-, ion-, and heat-balance; polarization data; gas bubble
effects; material compatibility; electrode reactions; cell geometry; etc.). It
is indicative that the inventor of the process was quite pessimistic about the
chances for success when this program started.

A year later, at the conclusion of the program, it can be said that the objectives
were largely met. The significant accomplishments were as follows:

> 1. Sufficient design data were generated, and detailed process
> steps were established for the scale-up of aluminum electroforming
> to commercially significant proportions. Admittedly, the process
> still has some shortcomings and is open for further improvements,
> nevertheless, its basic soundness was demonstrated, and further work
> can draw upon it.
>
> 2. The properties of the electroformed aluminum were established.
> The data show that this aluminum closely parallels the properties
> of the much used 2S aluminum, thus we obtain a highly useful metal.
>
> The laboratory specimens show that a properly run bath yields
> deposits of excellent surface characteristics (reproduction, re-
> flectance, etc.), and that deposition stresses are almost completely
> absent, in contrast to other electroforming processes. Thus, opti-
> cal quality components can be fabricated by this process, to a greater
> accuracy than by the conventional nickel electroforming or by any
> other currently known aqueous electroforming process.

3. The throwing power of the bath (ability to plate into deep recesses) was found to be so great that it nearly jeopardized completion of the program. Our 30" diameter nickel master would have produced good mirrors from a nickel or copper solution, but the ethereal aluminum solution plated, and thus keyed, into microscopically small pores preventing separation of the aluminum electroforms. These holes originated from almost unnoticeable pinholes in the epoxy master, reproducing as pinholes in the nickel master, due to bridging by the nickel deposit. In contrast, such holes reproduced as "keyed-in" peaks in the aluminum electroform. The quality of the master was not up to the standards demanded by the aluminum process, and since this could not be foreseen, four 30" mirrors were ruined before effective corrective actions could be taken. Once recognized, however, the formation of pinholes in the master can be prevented in future work by suitable resin formulation. This was demonstrated in the most recent spincast samples.

It should be noted that, in general, high throwing power is a very desirable characteristic in electroforming, since it lessens the requirement on anode conformance, gives more uniform deposit thickness and increases faithfulness of the surface reproduction.

An unforeseen consequence of the high throwing power was the unsatisfactory ability of the aluminum to bridge across the "grow-in" attachment borders. Thus, while the "gutter" ring was so well fitted to the master with conforming razor-sharp knife-edge that the assembly did not show any leakage of aqueous cleaning solution, the low viscosity, low surface tension ethereal solution, not only penetrated this knife-edge, but aluminum plated underneath, causing a faultline, instead of bridging across. Such faultlines are mechanically weak; the gutter can break away quite easily from the mirror, negating its purpose of acting as a buffer zone between the supported torus and the mirror. As a consequence, new attaching techniques will have to be worked out, e.g. by developing a conductive (silver-filled) resin, which would be applied at the point of contact of the knife edge as a bridge. Presently available conductive lacquers and resins are not sufficiently inert in the ethereal aluminum bath.

4. The rate of deposition of aluminum is highly satisfactory. Excellent deposits were obtained between 15 and 100 asf, and the upper limit was not established. Below approximately 10 asf, the coverage is increasingly unsatisfactory, the frequency of uncoated "islands" increase. At 20 asf, approximately 1 mil/hr deposit thickness is obtained (over 90% efficiency!), which is the same rate as nickel is generally electroformed. Due to the high ohmic resistance of the bath, the burden of cooling the solution increases as the current density is increased. Thus, increasing of the electrolyte conductivity presents a desirable avenue for future research.

5. The major shortcomings of the fabricated equipment were the filtration and agitation systems and the tank cover:

   (a) Filtration - In future designs, internal positive solution transport must be used instead of the alternate high and low pressure cycles in the filter. So much ether was evaporated (due to back pressure from the tanks) during each low pressure cycle, that continuous filtration during runs had to be abandoned. This was a difficult decision to make, since early in the program it was realized - and documented in several monthly and quarterly reports - that lack of good agitation and filtration resulted in rough, nodular deposits, which show through to the surface (orange-peely, locally distorted effect). Both final 30" diameter mirrors suffer from this shortcoming, while all the four smaller mirrors made in the laboratory under good filtration and agitation exhibit perfect surfaces.

   (b) The rotational movement of the cathode was too slow at approximately 2 minutes/rotation. The rotator was nitrogen driven, and due to gas leakage through the poppet valve of the reciprocating cylinder situated within the tank, the pressure had to be kept very low, 8 to 10 psi instead of the manufacturer's recommended 20 to 40 psi, to prevent over-pressurization of the electroforming tank. The consequence was the further decrease of agitation, already too low because of loss of filtration.

   In future work, direct motor-driven cathode rotation should be used.

   (c) Approximately 20 to 40 minutes were required to insert or remove the work from the bath, due to the numerous clamps holding down both the inner cover and the tank cover. Since this is quite inconvenient, and furthermore, prevents maintaining exacting residence times of preparatory steps, a more practical tank cover design would be desirable.

6. One of the most important aspects of this program was the repeated demonstration that aluminum electroforming is not only feasible and practical, but that it can be carried out in a safe manner. Inadvertent shorts, sparking, spillage, and solvent (ether) vapor leakage occurred on several occasions, just as these mishaps occur in conventional electroforming and plating rooms, without serious consequences. Labor, skilled in general electroforming practices, was easily trained to monitor the runs.

7. The two final mirrors submitted to NASA-Langley demonstrate
the basic soundness of the approach, practicability of the
technology, and the potential of the process. They also reflect
the shortcomings invariably present in "first try's".

The weight/surface area of these mirrors is highly attrac-
tive at 0.24 lbs/sq. ft. or $1.2KG/M^2$ (without torus).

The average slope errors for the copper-flashed and the all-
aluminum mirrors were 6.9 minutes and 9.4 minutes, respectively.
Mechanical polishing of the male nickel master, made necessary
in order to reduce "keying-in" of the aluminum electroform
(described previously), adversely affected the specularity and
optical accuracy of the master. It is axiomatic that a repli-
cated electroform can be no better than its mold. Some mecha-
nical interlocking still occurred after polishing, resulting
in more distortion of the all-aluminum mirror, as compared
with the copper-flashed aluminum mirror.

8. One of the most important achievements of this contract was
the development of a new separation technique. The separation
is an ever-present problem in any electroforming process, and
it rapidly increases in difficulty as the electroform thick-
ness decreases. As a matter of fact, it represents the most
serious stumbling block in the path of truly thin skins.
Electroforms must be deposited with certain degree of adhesion,
to prevent premature separation, solution slippage between the
surfaces, stress distortion, etc. Hitherto, this adhesion to
permanent substrates was invariably broken by some force such
as thermal expansion-contraction, localized air pressure, etc.

Removal of the outside air pressure in a vacuum chamber
was tried on the last mirror made under this contract. As soon
as the air pressure was reduced to about 100 microns Hg, the
thin aluminum skin (18 mils) popped off.

This technique should be applicable to any other thin skin
electroform, whether aluminum or a more conventional deposit.

4. LABORATORY INVESTIGATION

4.1   LITERATURE SURVEY

A survey of the published literature pertaining to the electrodeposition of
aluminum was made (see References 1 thru 4 and 6 thru 13). Personal contacts
with people knowledgeable in the field has enabled securing reports with limited
distribution on this subject, in addition to process details and observations.
Since the early work of Bunsen in 1854 (Ref. 12), a large amount of work has been
done on the electrodeposition of aluminum. The most successful of these processes,
the fused cryolite-alumina electrolysis for producing molten aluminum, is by its
very nature, not useful in electroforming. Because aluminum is much less noble
than hydrogen, it cannot be deposited from aqueous solutions.

The processes considered for electroforming are listed in Figure 4-1. Fused
salt baths, while much-mentioned, were deemed unsuitable because of the required
high temperature, resultant stress, probable attack of the optical surface of the
master, and because of their limited deposit thicknesses. The other baths, with
the exception of the $AlCl_3$-lithium hydride (Brenner-type) bath, were eliminated
on the basis of such criteria as unsubstantiated claims, safety considerations,
quality of deposit, reported poor results, failure to achieve greater than a few
mil thick deposits, etc. The choice for our scale-up studies was the ethereal
solution of aluminum chloride and lithium aluminum hydride (Brenner-type) as the
most promising for immediate results. This process was sufficiently understood
to be able to start at scale-up considerations instead of requiring fundamental
research and development.

Dr. Abner Brenner, of the National Bureau of Standards, inventor of the hydride-
type aluminum deposition process, generously offered his assistance. Dr. Leslie McGraw,
of Battelle Memorial Institute, studied the hydride process over several years,
and his comments have proved time-saving and valuable during the initial stages
of the program.

4.2   LABORATORY PLATING EQUIPMENT

The laboratory plating equipment was planned with the following in mind: to per-
mit a thorough investigation of all important process and design parameters; to
permit quick, simple modification of equipment; to be capable of simulating operating
procedures for large-sized electroforming; and to utilize stock items in place of
expensive specially-designed components.

4.2.1   GLOVE BOX

Since the plating bath reacts with oxygen, moisture, and carbon dioxide, a glove
box, in which all laboratory experiments would be performed in an inert atmosphere,
was secured. An airlock chamber, through which materials can be removed or in-
served without opening the glove box to the outside atmosphere, is attached to the
glove box (see Fig. 4-3).

Gas purge lines to regulate the flow of extra dry nitrogen (-75°F dew point) to the glove box were installed. Nitrogen exiting the glove box passed through an oil-filled bubbler and was vented directly into the hood. (Figure 4-3 illustrates this system.) Electrical connections within the glove box were soldered wherever feasible.

The glove box and auxiliary connections checked out leak-free.

4.2.2   FILTRATION APPARATUS

The ethereal bath attacks or softens most plastics, preventing the use of conventional plating room filters. A centrifugal-type filter pump, requiring priming would not be practical with this ethereal solution; therefore, a pressure-vacuum filter system with few moving parts was assembled, as described by Couch and Brenner (Ref. 4). It is composed almost entirely of glass, simplifying cleaning and drying. Connections were made with inert polyethylene tubing to increase flexibility. (See Fig. 4-2).

The filtration apparatus is composed of a vertical glass column, in which are supported layers of glass cloth covered with dry filter aid to prevent rapid clogging of the filter. A perforated aluminum disc was placed over this to prevent the glass cloth filter from "floating" upward. The column is covered with a glass cap sealed with a viton O-ring.

The plating bath was circulated through the filtration system by alternately decreasing and increasing the pressure within the filter chamber. The "higher" pressure cycle was provided by dry nitrogen gas (prepurified-grade, dew point -75°F). A vacuum pump was used to reduce the pressure for the alternate cycle. The subatmospheric pressure resulted in boiling in the filter, giving excessive ether volatilization losses. This difficulty was minimized by bleeding air directly into the vacuum pump to reduce the vacuum to a point where boiling did not occur in the filter chamber.

The filtration operation can be illustrated by referring to Figure 4-2. Plating solution enters the glass filter chamber during its reduced pressure cycle until a sufficient solution height is reached. The column was then pressurized to force the solution through the glass cloth filter bed. Glass check valves maintained flow in the proper direction. A 3-way solenoid valve connected to a current interrupter regulated the duration of each cycle, permitting automation of the filtration process.

The intermittant filtration during laboratory operation kept the volatilization losses within tolerable limits; however, the close watch needed with this unit was not practical for scaled-up operation. The filtration unit for the scaled-up equipment was designed so that the pressure varied between 6-10 psig and atmospheric pressure (high and reduced pressure cycles, respectively). These conditions were first checked out in the laboratory with complete success, operationally and with respect to ether losses.

4.3 COMPOSITION OF THE ALUMINUM PLATING SOLUTION

The hydride-type bath consists basically of an ethyl ether solution of anhydrous aluminum chloride and lithium hydride (or lithium aluminum hydride). Dr. McGraw recommended the use of 3 to 3.5 M aluminum chloride concentration.

Our work established that a 3.4M $AlCl_3$ - 0.4M $LiAlH_4$ - diethyl ether solution represented the optimum composition for the hydride-type bath.

4.4 PREPARATION OF THE PLATING SOLUTION

Various feasible procedures for preparing the plating solution for electroforming the 30" diameter mirrors were investigated. The slow, careful addition of ether to $AlCl_3$ resulted in potentially hazardous localized overheating, even when external cooling was employed. On occasion, the highly exothermic heat of solution (580 calories per gram) caused an almost uncontrollable, runaway temperature rise. Slow additions of $AlCl_3$ to ether was the most controllable procedure for preparing the plating solution. This procedure, with adequate cooling to minimize ether volatilization losses, was adopted for the preparation of the solution to electroform the 30" diameter aluminum mirrors. Subsequent addition of $LiAlH_4$ posed no problems, although evolution of gas occurred at the start. This is normal and no cause for concern.

In preparing each batch, glass tanks and laboratory glassware containers were used to eliminate plating bath contamination. Ether was added slowly through a teflon plug-glass stopcock valve. Agitation, during solution of solids, was provided by a glass-enclosed magnet in the mixing tank, with the motor and magnetic drive located outside the glove box. This worked satisfactorily and permitted use of less costly, non-explosion proof equipment.

All of these processes yielded equally suitable electrolytes. In evaluating these processes, it appears that external cooling during bath preparation must be employed as a safety measure when preparing large volumes of plating solution. The use of aluminum lithium hydride instead of Brenner's original lithium hydride formulation more than doubles bath life (Ref. 8).

4.5 PLATING BATH LIFE

4.5.1 IN STORED CONDITION

One plating solution was sealed and stored for a month after preparation in glass containers. Electroforms deposited from this "aged" solution did not differ in any respect from deposits from a freshly prepared solution. A tightly sealed plating solution has a long life, measurable at least in months.

4.5.2 DURING DEPOSITION

The solution was permitted to go to exhaustion during a planned series of deposition runs to establish bath life in use. After 5 weeks intermittant use, the plating bath showed signs of exhaustion: the stresses noticeably increased, and the ductility of the deposits decreased. Increase of absorbed water content was particularly detrimental to the bath.

The second plating solution, 1½ liters volume, was operated under conditions of continuous test and maintenance. After initial deposition runs, 2 liters of a like-composition solution were added to scale up this bath, and more runs were conducted; the volume was further increased to 5½ liters. At the end of thirteen electroforming runs, 2 more liters of solution were added, and a 7" diameter paraboloidal mirror was deposited from the now 7½ liter solution. Altogether, approximately 44% of the aluminum content of the 7½ liter bath was depleted when this phase of the study ended. The deposits remained low-stressed and very ductile throughout the runs. Lithium aluminum hydride additions were made periodically to maintain the initial concentration.

4.6 ELECTRODES

4.6.1 ANODE

Commercially pure (2S or 1100) aluminum anodes were used during deposition to maintain bath purity. The anode geometry was a flat plate even with the hemispherical or parabolic electroforms. The throwing power of the electrolyte (ability to plate geometrically remote regions) proved to be surprisingly high. However, a conforming anode was used for the 30" diameter mirrors to insure greater uniformity of deposit thickness. The anode must be shrouded in glass cloth to contain the black anode slime, prevent its contamination of the plating solution, and reduce the amount of pore-clogging sediment to the filter. Glass cloth was ascertained to be superior to various alternative materials cited in Ref. 4 (i.e., alundum, cellulose pulp, polyethylene, felt paper, various rubbers), as regards compatibility with the plating solution, sufficient porosity for deposition, strength, non-deterioration, pliability, availability in various sizes and in cost.

4.6.2 SUBSTRATE

Aluminum electroforms have been deposited successfully on nickel, copper and brass substrates in the laboratory. The primary emphasis, however, has been with electroformed nickel substrates to simulate the 30" diameter electroformed male nickel master. Accordingly, various sizes of flat electroformed nickel tools were utilized for deposition. Subsequently, paraboloidal (5" and 7") and hemispherical (2½") electroforms were deposited on electroformed or plated nickel masters to simulate the ultimate geometry and substrate material. No difficulties were experienced in any of these runs.

4.7   PRE-TREATMENT OF SUBSTRATE

Successful electroforming requires that the deposit exhibit sufficient adhesion
to prevent premature parting during deposition, and still permit easy separation
at the conclusion of the run, so that the electroform need not be pried off and
thus distorted.

For the initial ("shakedown") run, the cathode substrate received no pre-treatment.
The subsequent deposit, although extremely thin, peeled away prematurely from the
substrate. Many complex substrate treatment procedures are cited in the litera-
ture (Ref. 4, 8, and 9). A simple procedure utilizing an oleic acid solution was
developed in the laboratory and is used in conjunction with a thin chemically-
reduced silver film applied to the substrate. The drawback of this method is the
adherence of the silver film to the electroformed aluminum; however, it permitted
distortion-free separation of the soft aluminum deposit, which is quite significant
when considering the problems generally encountered in pulling off the much more
rugged nickel electroforms from their masters. After about a month's storage in
a desk drawer, the silvered surfaces showed signs of tarnish. Methods to remove
the adhering silver film without damaging the geometry and reflectivity of the
aluminum substrate or omitting the silver film from the procedure were investi-
gated with only moderate success.

4.8   FILTRATION

Couch and Brenner (Ref. 4) stated that the plating solution does not require fre-
quent filtration, since the insoluble impurities and black anode slime settle to
the bottom of the laboratory cell and usually do not cause rough deposits. This
contradicts the data from our runs, in which filtration was omitted. Rough nodu-
lar deposits were obtained without filtration, when the aluminum electroform
thickness exceeded 0.008 to 0.010-inch without current reversal, or exceeded 0.012
to 0.014-inch with current reversal. With repeated filtration during deposition,
the number and size of nodules sharply decreased when the proper current reversal
cycle was employed, even at deposit thickness in excess of 0.030-inch. This is
extremely important in our program, since the nodules on the rear surface show
through to the front.

4.9   AGITATION OF THE PLATING SOLUTION

A glass-covered magnetic stirrer was used to provide mechanical agitation of the
plating solution during several deposition runs. This agitation was observed to
stir up anode sediment, making filtration more difficult, and resulting in a
roughened deposit. Agitation was reluctantly omitted from subsequent deposition
runs.

In electroforming the 30" diameter mirror, where agitation was considered manda-
tory, the design called for the filtered solution, returning from the filter, to
impinge directly on the male nickel master, and for a slow rotational motion of
the male nickel master, which would result in gentle secondary agitation and serve
to make the deposited thickness more uniform.

4.10   CURRENT DENSITY

The character of deposit for a range of current density (0 to 140 asf) was in-
vestigated utilizing General Electric's proprietary Stress Cell (Ref. 14). The
deposit from just one run permitted evaluation of the entire current density range
cited above. Bare spots and some peeling were noted at less than 10 asf, whereas,
at high current densities, the deposit turned rough. A relatively wide current
density range (15-100 asf) was found to be suitable for our requirements. Alumi-
num electroforms deposited within this range (generally about 20 asf) were highly
satisfactory. This represents a deposition rate equivalent to 0.001 inch alumi-
num deposited per hour.

4.11   CURRENT REVERSAL

The smooth deposits which can be achieved with current reversal have been reported
by Couch and Brenner (Ref. 4). Our laboratory investigation came to the same
conclusion. Short deplating cycles were not effective. A 294-second plating,
6-second deplating time cycle produced excellent results for our process conditions.
The 6-second (2% time) deplating cycle proved optimum in preventing nodule growths,
while not greatly reducing the overall plating efficiency.

The effect of not using current reversal in electroforming "thick" deposits is
the increased tendency to form nodule growths which show through the front surface.
This is shown pictorially in Figure 4-8a, where close examination of the bright
mirror surface reveals image distortion at the points of nodular growth.

4.12   DEPOSITION TEMPERATURE

Our laboratory cell, contained within a glove box, had no provision for cooling,
therefore, some rise in temperature resulted due to resistance heating. The
character of the electroform deposit was not affected within this $\pm$ 15°C tempera-
ture fluctuation.

The plating solution boiling point proved much higher than anticipated. The
equilibrium temperature of one deposition run was 50°C, and although the volatili-
zation losses were high, boiling did not occur. It should be noted that pure
diethyl ether boils at 35°C. A reflux condenser was incorporated into the final
design, to minimize ether volatilization losses.

4.13   ELECTRODE SPACING

The cathode-anode spacing for the initial series of aluminum depositions was
4½ inches. The electrode spacing was gradually decreased from 4½ inches to 2 inches
to lower the resistance. No differences were noted in the character of the alumi-
num deposited on the various metal substrates (i.e. nickel, copper and brass).
Closer spacing than 2 inches did not appear advisable because an occasional long,
slender tree growth could short out the cell.

4.14   CELL VOLTAGE BALANCE

An electrolyte resistivity of 90 ohm-cm was measured for a freshly-prepared room
temperature plating solution. Polarization potential was measured at approximately
0.6 volts as part of the over-all cell voltage balance (see Fig. 4-5). Close to
100% cathode efficiencies were obtained.

4.15   CELL VOLUME AND GEOMETRY

In the laboratory, the initial plating solution volume of 1½ liters was scaled
up successively to 3½, 5½ and 7½ liters (approximately 2 gallons). The cell geo-
metry was varied: 2½" x 4¼" x 6", 6" x 7½" x 2½", 6¼ x 9½" x 10" and 6" diameter
x 3½". The electroforming operating conditions remained predictable during the
cell volume, geometry, and specimen size scale-ups.

4.16   THICKNESS

Conner and Brenner (Ref. 4) recommended the addition of methyl borate to the plating
solution to attain deposition thickness. In the laboratory cell, aluminum electro-
forms have been deposited to thicknesses of 0.029" for a flat plate, and in ex-
cess of 0.030" for a 7" diameter paraboloidal mirror without bath additives or any
difficulty. This was encouraging, since all the additives tried increased the
stress levels in the deposits.

4.17   THROWING POWER

The throwing power of the aluminum plating solution was excellent. Accordingly,
several parabolic and hemispherical electroforms were deposited in the laboratory
cell using a flat (non-conforming) anode. Thickness variations in the 7" diameter
electroformed paraboloidal mirror did not exceed ± 0.001 inch with the non-conforming
anode.

4.18   STRESSES

The effects of current density and temperature variations on stress were surprisingly
small. Measurements were taken with GE's proprietary Stress Cell. Electrodeposi-
tion stresses were less than ± 80 psi (resolution limit at the instrument setting).
Stress increased markedly when the bath moisture level increased. Accordingly,
tight process control was indicated and maintained.

4.19   POWER FAILURE

Anticipating possible power failure during the deposition of the 30" diameter alu-
minum mirrors, the consequences and possible corrective procedures were evaluated
in the laboratory cell. Power shut-downs or removal of the electroform from the
plating solution caused delaminations in the deposit. Corrective procedures, such
as anodic etching, oleic acid activation, etc., proved ineffectual, so that an
auxiliary power supply was incorporated into the equipment design, which would

maintain the master cathodic during failure of the plating current. This safeguard was tried out in a laboratory run, when the current density was reduced to a trickle for 8 hours. The deposition run was later resumed, and the resultant electroform was devoid of laminations.

4.20   PLATING BATH CONTROL

After 5 weeks of intermittent use, the initial aluminum plating bath showed signs of exhaustion. The plating stresses noticeably increased, and the ductility of the deposits decreased from Run 10 on. This was found to be due to increase in water content.

Absorbed water was detrimental to the plating solution. A very simple monitoring procedure was suggested by Dr. L. McGraw and checked out in the laboratory as an effective control device. Increased electrode depolarization time indicated an increased moisture content in the plating solution. This was easily measured with an oscilloscope, and a pictorial record was secured (see Figures 4-4a, b, & c). The addition of hydride compensated for the increased moisture in the bath. The effect is shown dramatically in Figures 4-6a and b.

4.21   DEPOSITED ALUMINUM ELECTROFORMS

4.21.1   FLAT PLATES

A series of 14 plating runs were performed in the laboratory to study bath parameters. These runs varied in duration from 12 minutes for a "shakedown" run to a 33.5 operational hours run in 4 days, with deliberate interruptions simulating various breakdowns. Deposits up to 0.016" thick were prepared in this series. Many of these runs were conducted at other than optimum conditions to secure necessary data. All pertinent data for these runs are presented in Table 4-1.

In further runs, several flat plates were electroformed over electroformed nickel tools which had been plated on static-poured GE epoxy (spincast formulation). These plates ranged in size from 2 square inches to 24 square inches, and in thickness from several thousandths to 0.029-inch. The plating solution was contained either within a circular glass tank or two different-sized rectangular glass tanks during these runs. The electroformed plates lacked visible distortion, were highly reflective, and exhibited excellent replication of the master surface. The 0.029-inch aluminum plates were used to machine physical property test specimens, described in a later section.

4.21.2   5-INCH DIAMETER PARABOLOIDAL MIRROR

An electroformed male nickel master was secured to attempt electroforming a paraboloidal aluminum mirror by bringing it through the entire planned process (pretreatments, estimated residence times, design geometry, etc). The data from this run were regarded as an essential feature of our laboratory study. The result (a 5" diameter paraboloidal mirror) was highly pleasing, and is presented pictorially in Figures 4-7a and b.

The surface replication was excellent. The reflected images (i.e. florescent lights, camera, ceiling beams, etc.) indicate a distortion-free surface resulting from the process. A conforming anode was not used, therefore, the throwing power appeared surprisingly high (uniform thickness!). Nonetheless, a conforming anode was used for the large mirrors. After about a month's storage in a desk drawer, the silvered surface showed some tarnish.

4.21.3   2½-INCH DIAMETER HEMISPHERICAL MIRRORS

A 2½" diameter hemispherical electroformed male nickel master was used to electroform two 2½" diameter mirrors. The results were comparable to that for the 5" diameter paraboloidal mirror and are presented pictorially in Figures 4-8a and b. The reflected images indicate a very good replication.

In Figure 4-8a, the left mirror was made without periodic reverse cycling, therefore, increased nodulation occurred on the back surface. On one of these mirrors, the silver was removed by gentle mechanical polishing (impregnated cotton), but this polishing ruined the specularity.

4.21.4   7-INCH DIAMETER PARABOLOIDAL MIRROR

The 1½-gallon plating solution was brought up to 2 gallons to accommodate a 7" diameter male electroformed copper master, coated with nickel. The quality of the nickel surface was rather poor with some scratches, nicks, pits, and show-thru of copper visible; however, the geometry was acceptable. The master was a reject specimen, secured at no cost, through the courtesy of Bart Manufacturing Corporation. Despite these shortcomings, the scale-up data which would be gained justified this run. These defects are less noticeable on a convex surface and are not readily visible in the photograph (Fig. 4-9a).

The results were, once more, highly pleasing, although faithful replication of the defects marred the aesthetic appeal. The 7" diameter paraboloidal mirror is shown pictorially in Fig. 4-9b. The reflected images (florescent lights, camera, ceiling beams, the authors of this report, stripes in tie, miscellaneous objects) indicate surface free from visible geometrical distortions. A conforming anode was not used, but the uniformity of the deposit was good. The relatively smooth back surface is shown in Fig. 4-9c.

The thickness of the deposit exceeded 0.030 inch.

One important lesson learned from all these mirror runs was that the larger nodules on the rear surface show through to the front, causing an orange peel effect.

4.22   DISPOSAL OF PLATING SOLUTION RESIDUES

To date, no attempts have been made to dispose of any sizable quantities of plating solution. However, disposal of small quantities of plating solution (about 2 liquid ounces each bottle) used for compatibility studies, solution adhering to the electrodes, etc., have been performed without difficulty. The aluminum electroforms in the laboratory were rinsed thoroughly in fast-running water after removal from the bath to react all adhering plating solution. A 2-gallon jar of water was kept in the hood for quick immersion of the anode assembly. The small quantity of plating solution, added slowly in small increments to the 2 gallons of water, did not heat up the water excessively. Concurrently, portions of the plating solution were left exposed in the hood to reduce the volume through evaporation and react slowly with the moisture and oxygen of the air. For the small quantity of solution, this latter technique was not necessary, although this would be employed for disposal of the bulk of the laboratory plating solution, when necessary.

4.23   MATERIALS COMPATIBILITY

It was known that glass or glass-lined equipment would be suitable for electroforming the 30" diameter mirror, but because of its high cost and fragile character, less expensive alternates were sought. Also, the use of some maskant is required in electroforming. Therefore, a concurrent investigation of the compatibility of various materials, with the liquid and vapor phase of the plating solution, was conducted. The results are summarized in Table 4-2. The data from these tests formed the basis of materials design for the scaled-up aluminum electroforming equipment.

FIGURE 4-1

ALUMINUM - DEPOSITING BATHS

1. Fused Salts: $AlCl_3$ - NaCl
                 $AlCl_3$ - LiCl
                 $AlBr_3$ - NaBr
                 $AlBr_3$ - KBr

2. Al-Halide - quaternary ammonium salts, etc., dissolved
   in ethyl bromide.

3. Alkyl aluminum halides dissolved in ethyl ether.

4. $AlBr_3$ - ethyl bromide - benzene.

5. Aluminum alkyls dissolved in toluene or xylene.

6. $AlCl_3$ - ethyl pyridinum bromide in benzene or toluene.

7. $AlCl_3$ - LiH (or $LiAlH_4$) - ethyl ether.

FIGURE 4-2

FILTRATION SYSTEMS TO PLATING BATH
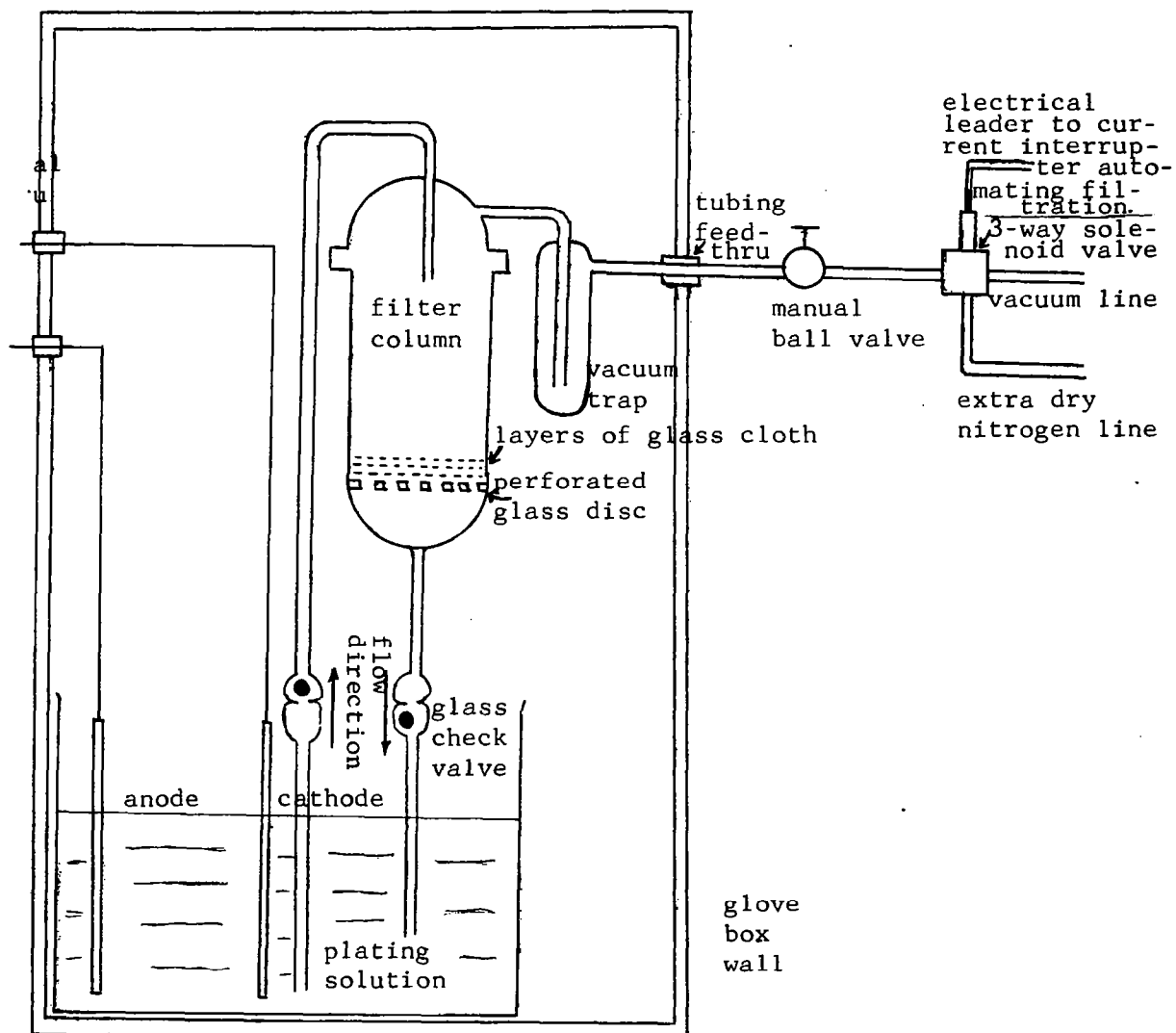
FIGURE 4-3 : GLOVE BOX PURGE SYSTEM

vents directly
into hood

gas
bubbler
(thru
oil)

valve

shatterproof viewing glass

glove
ports

glove box
and stand

airlock chamber

2-stage
pressure
regulator

gas flow meters
with needle valve

nitrogen line
to filter

cylinder
extra-
dry
nitrogen
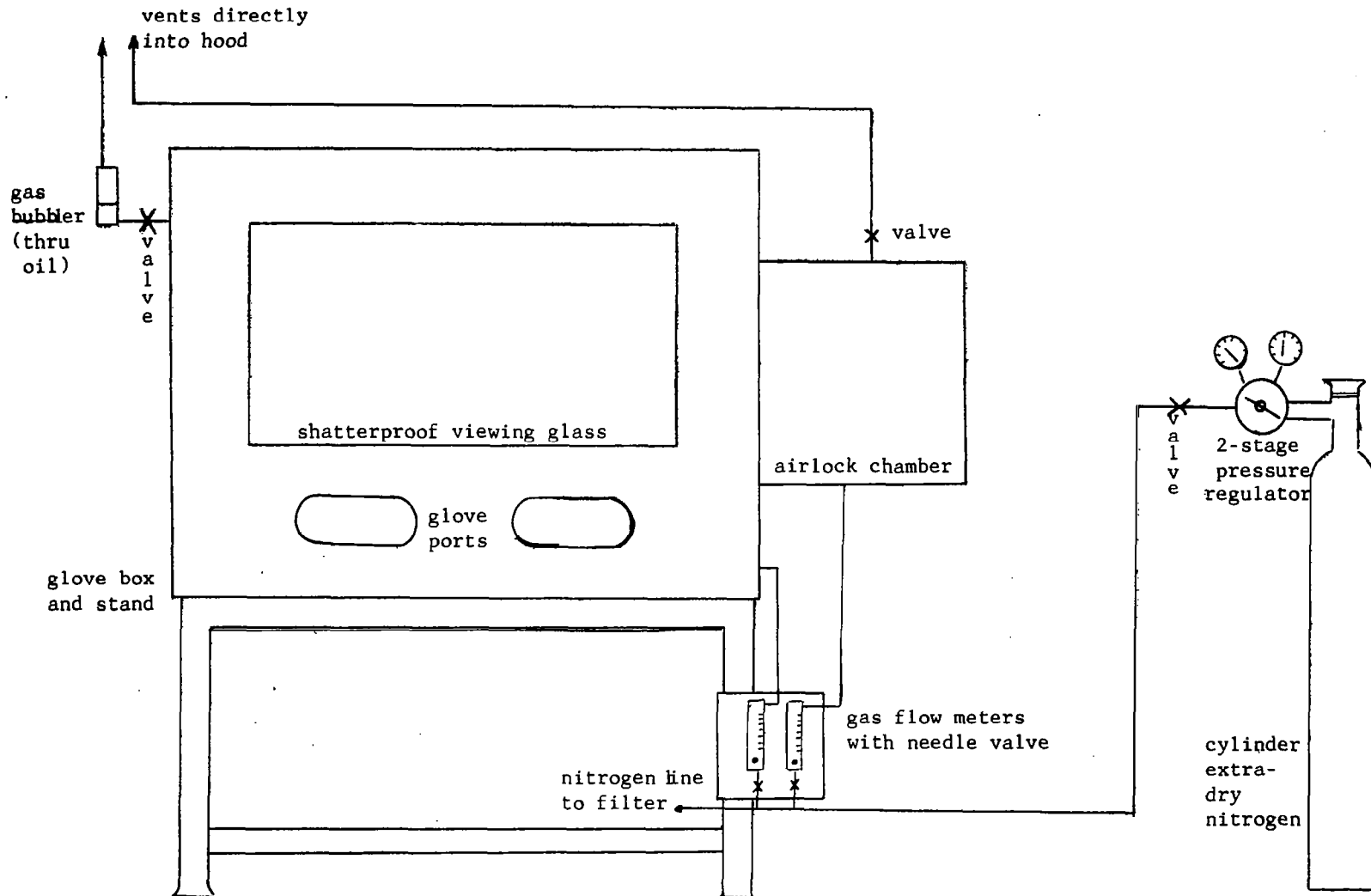
FIGURE 4-4- DEPOLARIZATION TIME
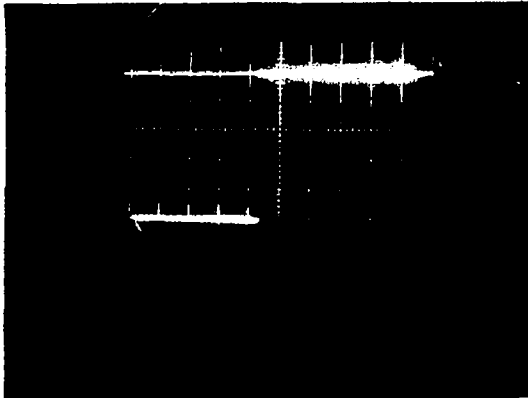
Fig 4-4a - Time photograph of oscilloscope voltage measurement at the interruption of Run 12. Vertical scale: 5 volts/major division. Horizontal scale: ½ second/major division. Power interruption dropped the voltage from 25 to 0 volts instantaneously.
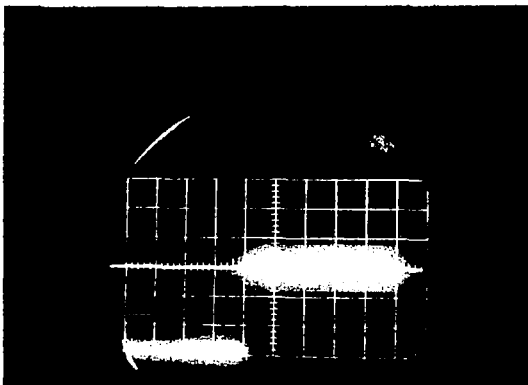
Fig 4-4b - Time photograph of oscilloscope voltage measurement at the interruption of Run 13. Verticale scale: 5 volts/major division. Horizontal scale: ½ second/major division. Power interruption reduced voltage from 15 volt. Note: voltage trace exhibits a discernable difference from zero volts.
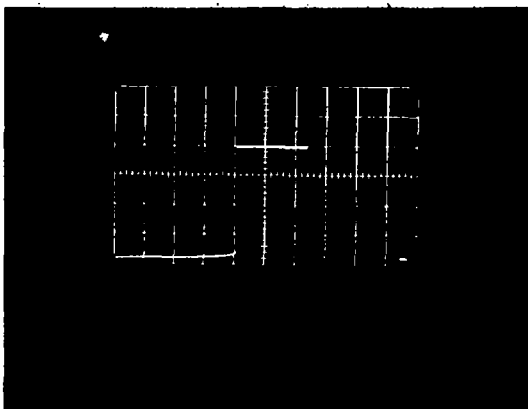
Fig 4-4c - Time photograph of oscilloscope voltage measurement performed at the interruption of Run 14. Vertical scale: 2 volts/major division. Horizontal scale: 2 sec/major division. A black flakey deposit now occurs. Note: voltage did not return to zero within the 8 seconds time recorded in the photograph.

FIGURE 4-5: OVERVOLTAGE CURVE
for Bath #2 (unused)-3.4M AlCl₃, o.4M LiAlH₄ in
diethyl ether at 23½°C
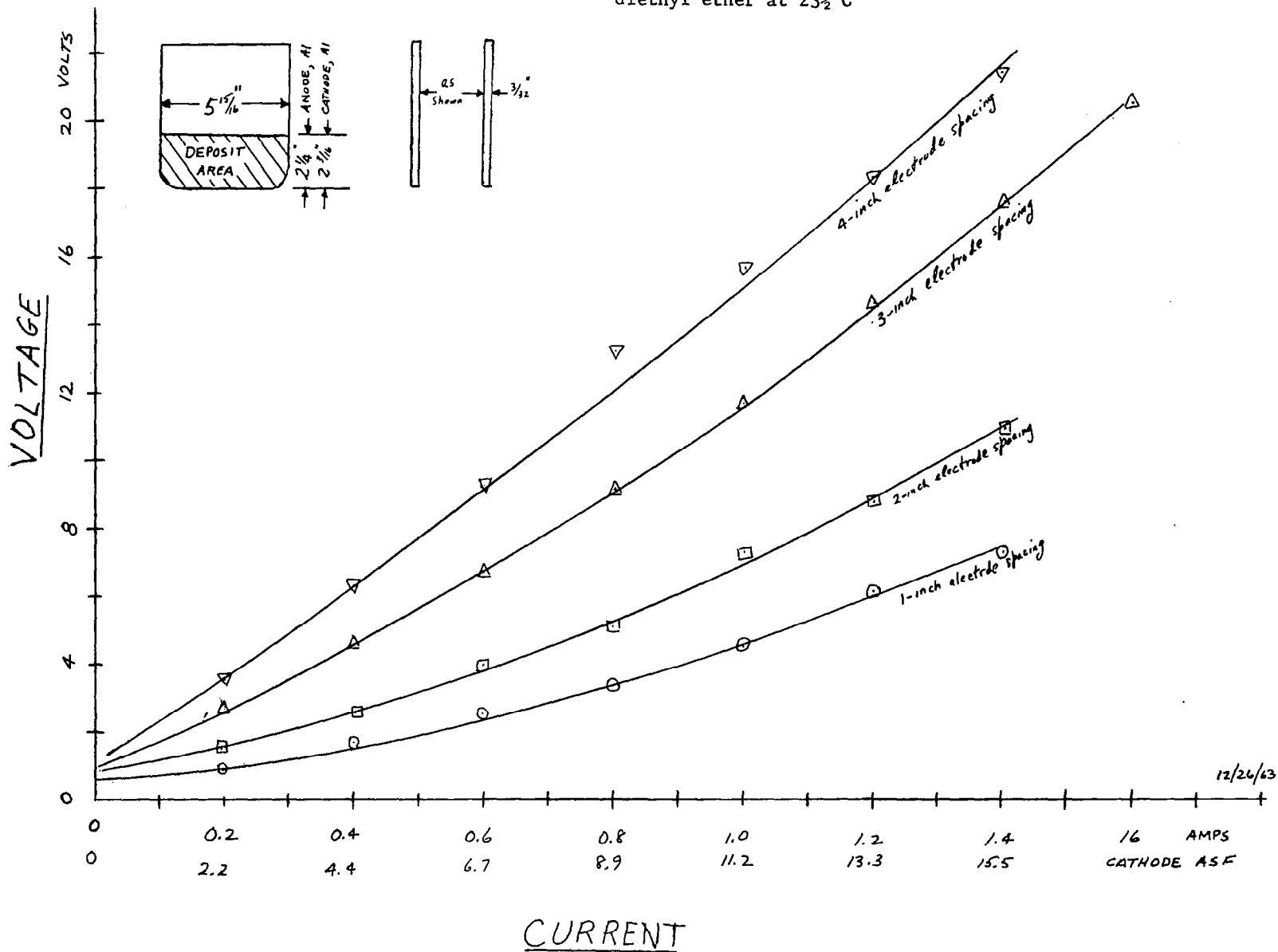
FIGURE 4-6

DEPOLARIZATION TIME - INITIAL RUN
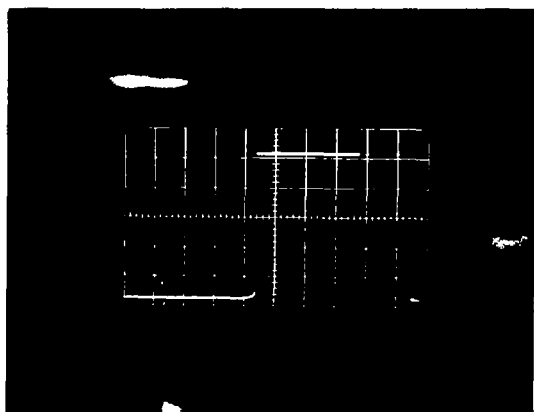
Figure 4-6a - Time photograph of oscillo-scope voltage measurement at the inter-ruption of a psuedo-deposition run in the "exhausted" initial plating bath. Ver-tical scale: 2 volts/major division. Horizontal scale: 1 second/major divi-sion. Power interruption dropped vol-tage to 0.6 volt (polarization voltage). NOTE: Voltage did not return to zero within the 4½ seconds recorded in the photograph.
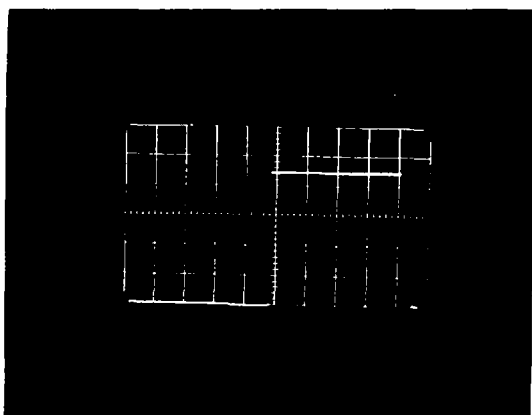
Figure 4-6b - Time photograph of oscillo-scope voltage measurement at the inter-ruption of a psuedo-deposition run after 0.1M LiAlH$_4$ was added to the initial plating bath shown above. Vertical scale: 2 volts/major division. Horizontal scale: 1 second/major division. Power inter-ruption dropped voltage to zero. Deposits from this bath were again low stressed and ductile.

FIGURE 4-7

5" ELECTROFORMED ALUMINUM PARABOLOIDAL MIRROR
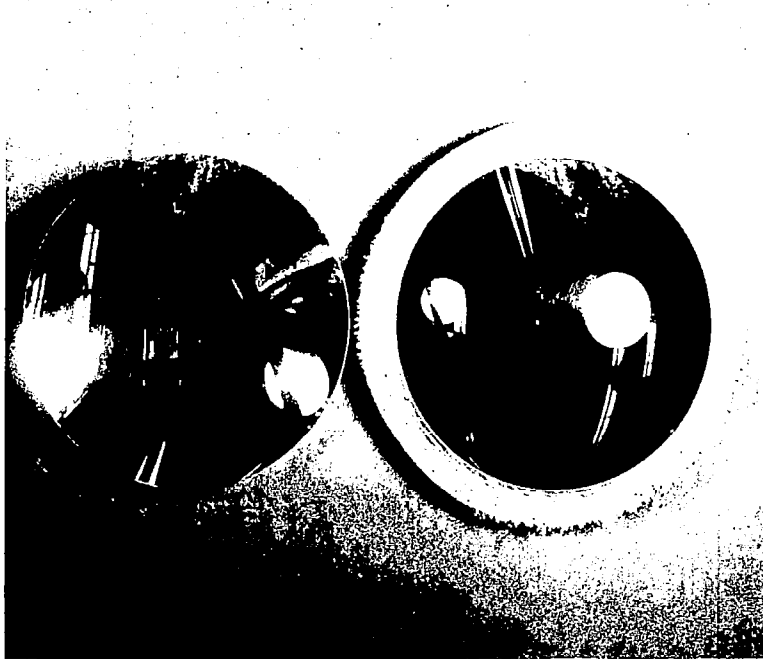
Figure 4-7a: First electroformed
aluminum paraboloidal mirror (5")
on left; nickel master from which
mirror was replicated on right. Note
the undistorted reflections in the
mirror (i.e. camera, florescent
lights, flat rod held in hand, fin-
gers, etc). Mirror surface is as
removed from nickel substrate - un-
washed, unpolished, etc.
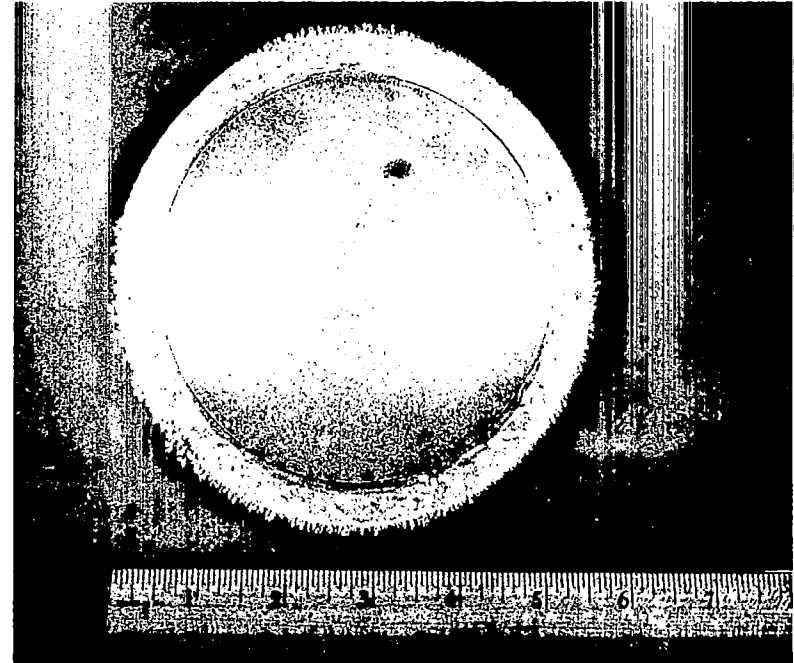
Figure 4-7b: Five-inch electroformed
aluminum mirror (see Fig. 4-7a) prior
to removal from nickel substrate. Note
the smooth deposit and lack of nodular
growths over the mirror portion of the
deposit. Edge growths ("trees") are re-
latively minor as compared to nickel
or copper electroforming.

FIGURE 4-8

2½" ELECTROFORMED ALUMINUM HEMISPHERICAL MIRROR

Figure 4-8a: First electroformed
aluminum hemispherical mirrors (2½"
diameter). Bottom left mirror is as
removed from electroformed nickel
master above. Bottom right mirror was
mechanically polished. Note undis-
torted images (i.e. camera, florescent
lights, ceiling poles and beams, etc).
Haze lines about incandescent lamps
appear in mirror and master, demonstrating
excellent replication.
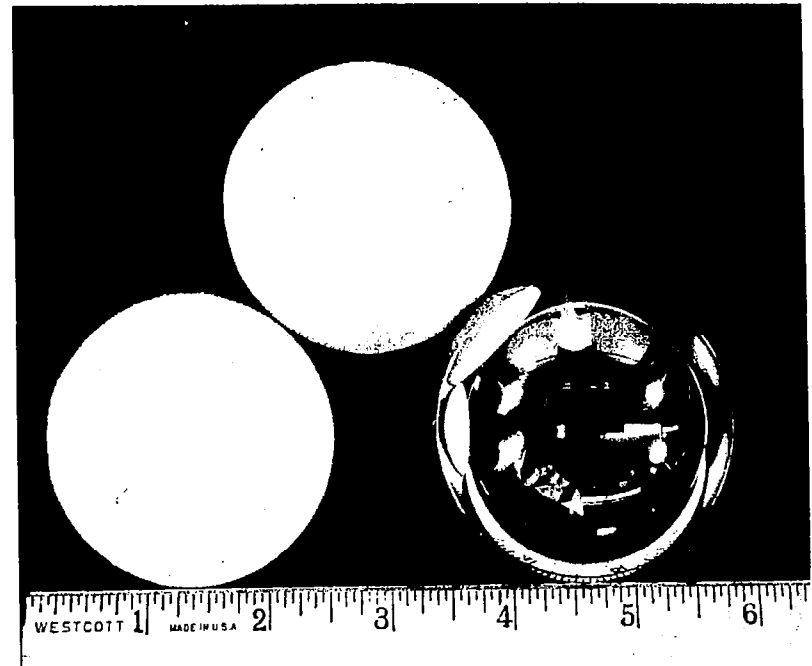
Figure 4-8b: Back surface of the 2
hemispherical mirrors. Note the lack
of nodular growths on the deposited
surface of the upper electroform.
Electroform on bottom left, where
current reversal was omitted, did have
small nodular growths resulting in
"show-thru". Electroformed nickel
master is also shown.

FIGURE 4-9

7" ELECTROFORMED ALUMINUM PARABOLOIDAL MIRROR
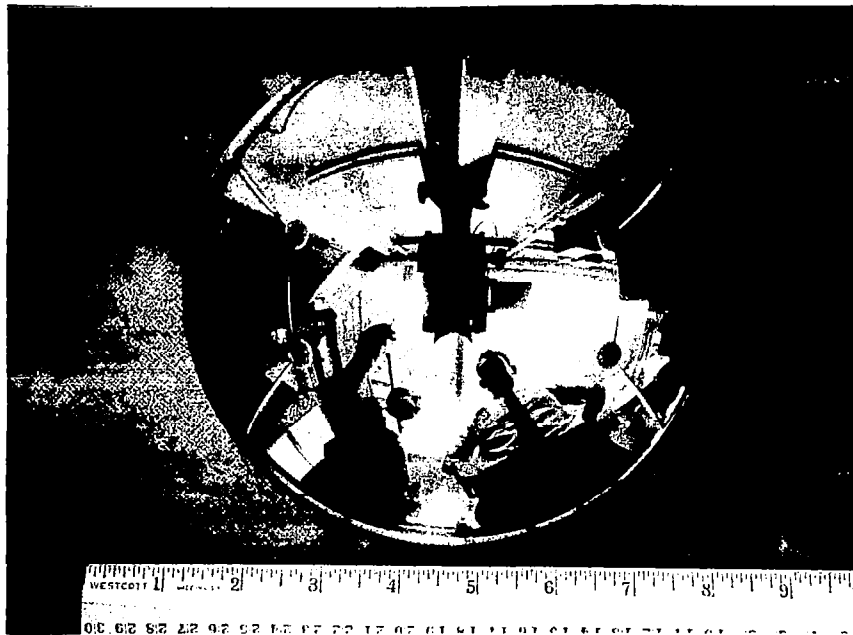
Fig. 4-9a - Male Nickel Master: Used to electro-
form the 7" aluminum paraboloidal mirror shown in
Fig. 4-9b. Certain imperfections in the master
are visible at 10 o'clock position of this photo.

FIGURE 4-9

7" ELECTROFORMED ALUMINUM PARABOLOIDAL MIRROR
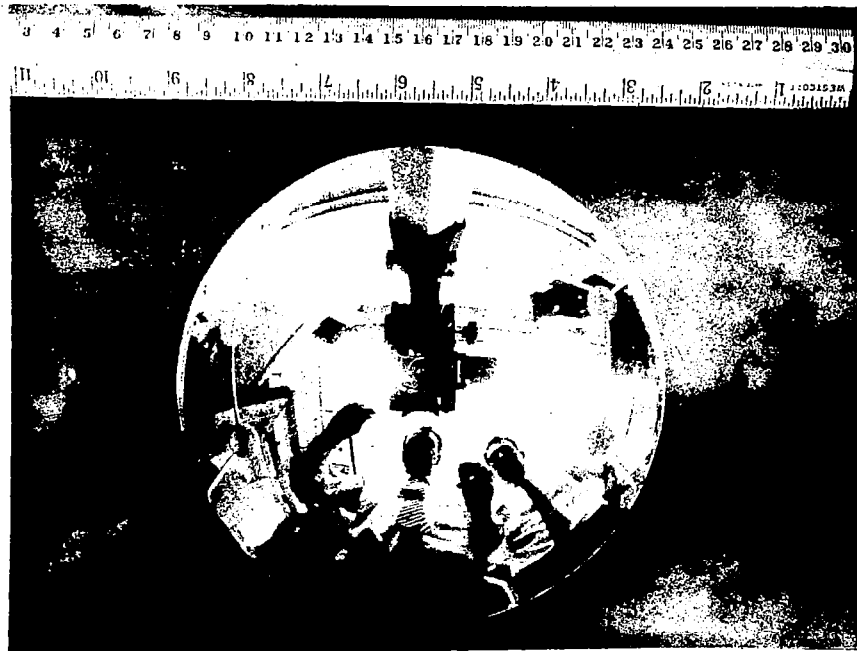
Fig. 4-9b - Mirror: 7" mirror replicated from the
nickel master shown in Fig. 4-9a.   The aluminum
mirror was electroformed using design parameters of
the 30" mirror.   Note the undistorted reflections
in the mirror (i.e. camera, florescent lights,
ceiling poles, beams, images of Dr. F. Schmidt and
I. Hess, facial detail, stripes of tie, etc.).
Mirror surface is as removed from nickel substrate.

FIGURE 4-9

ELECTROFORMED ALUMINUM PARABOLOIDAL MIRROR
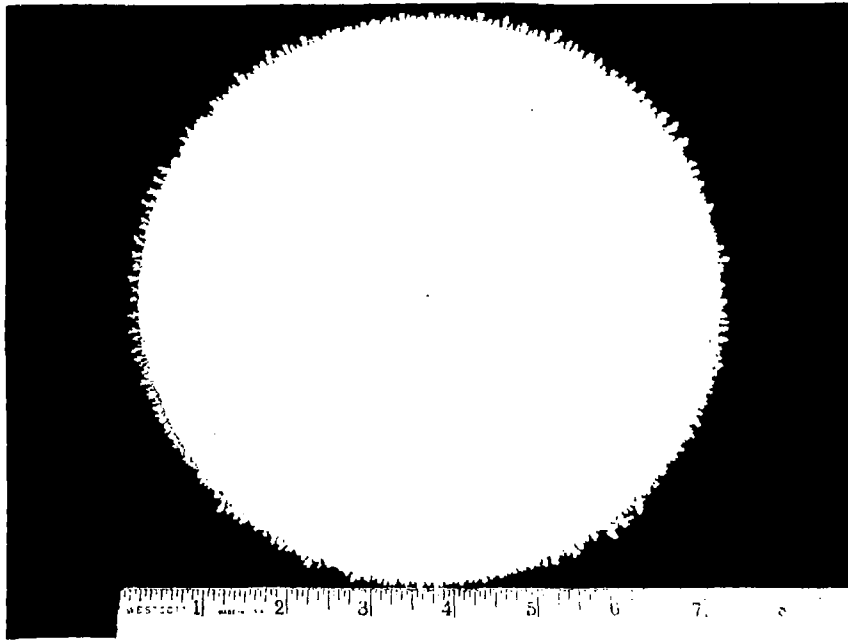
Fig. 4-9c - Back Surface: Back surface of the
7" electroformed aluminum paraboloidal mirror prior
to removal from nickel master. Note the small edge
growths ("trees") despite the 0.030-inch thick deposit.

BATH No. 1 (prepared 23 November 1963) 1.6 liter 3.4M $AlCl_3$, 0.40M $LiAlH_4$ diethyl ether solution.

Run No.	Date	Oper Time (hrs)	Cathode	(i) Anode	ACTUAL Cathode Area (in²)	Anode Area (in²)	NOM. Deposit Area (in²)	Current (amps)	Voltage (volts)	NOM. Current Density (asf)	(a) Effect. Current Density (asf)	Cathode Back Surf. Protection
1	11/26/63	0.2	Nickel(f)	Aluminum	(b)	(b)	(b)	0.50	-	(b)	(b)	yes
2	11/26/63	1.0	Copper	Aluminum	(b)	(b)	(b)	0.55	-	(b)	(b)	yes
3	11/27/63	1.0	Nickel(f)	Aluminum	5.17	6.5(d)	6	0.75	-	18	20.6	no
4	12/2/63	3.5	Nickel(f)	Aluminum	8.19	9.6(d)	7.5	1.0	-	19.2	17.6	no
5	12/3/63	1.0	Brass	Aluminum	(b)	(b)	(b)	0.55	-	(b)	(b)	yes
6	12/3/63	10.5	Nickel(f)	Aluminum	3.82	6(d)	3.75	0.60-.95(e)	-	23-36(e)	23-36(e)	no
7	12/5/63	10.0	Nickel(f)	Aluminum	8.17	9.6(d)	7.5	1.0-3.8(e)	-	19-73(e)	22-86(e)	no
8	12/6/63	6.0	Copper	Aluminum	8.84	9.6(d)	9.0	1.2-1.0	-	19-16	19-16	no
9	12/9/63	7.0	Nickel(f)	Aluminum	5.04	6.5(d)	6.0	0.85	-	20.4	24.3	no
10	12/10/63	7.0	Nickel(f)	Aluminum	5.33	9.6(d)	6.75	0.90	-	19.2	24.3	no
11	12/12/63	5.0	Nickel(f)	Aluminum(2s)	11.2	14.0(d)	12.0	1.50	24.5-30(g)	18.0	19.2	no
12	12/17/63	1.25	Nickel(f)	Aluminum	(b)	(b)	(b)	1.0	27.5	(b)	(b)	yes
13	12/17-20/63	33.5	Nickel(f)	Aluminum(2s)	6.71	7.58	7	0.70	7.7	11.5	12.0(h)	yes
14	12/26-30/63	20.0	Nickel(f)	Aluminum(2s)	6.25	7.58	7	0.70	8.6	11.5	12.8	yes

FOOTNOTES:

(a) Includes stray deposits.

(b) GE Stress Cell Run.

(c) Total current during GE Stress Cell Run.

(d) Estimated

(e) Bath heated during night, current increased due to lower IR drop.

(f) Nickel sheet, commercial grade, US gauge No. 24.

(g) Solution volume fluctuated due to evaporation. Ether added periodically to restore to original volu

(h) Adjusted for deplating cycle.

(i) 6061 Aluminum used for anode except where otherwise indicated.

TABLE 4-1

Electrode Spacing (in)	Electrolyte Geometry (in³)	Equil Temp (°C)	POWER USED		Current Reversal (if used)				Cathode Pretreatment
			This Run (AH)	Cumulative (AH)	Plating Cycle CD (asf)	Time (sec)	Deplating CD (asf)	Time (sec)	
(b)	6"x7½"x2½"	-	0.10	0.10	0.50a(c)	150	0.75a(c)	10	Solvent cleaned, 100% oleic acid
(b)	6"x7½"x2½"	29	0.55	0.65	no current reversal				Cleaning sol'n, 10% oleic
4½	as above	32	0.75	1.40	no current reversal				Cleaning sol'n, 10% oleic
4½	as above	38	3.50	4.90	no current reversal				Cleaning sol'n, 10% oleic
(b)	as above	29	0.55	5.45	no current reversal				Cleaning sol'n, 33-1/3% oleic
4½	as above	40	8.1	13.55	no current reversal				Cleaning sol'n, 33-1/3% oleic
4½	as above	45	17.0(f)	30.55	no current reversal				Cleaning sol'n, 20% oleic
4½	as above	45	6.8	37.35	no current reversal				Cleaning sol'n, 20% oleic
4½	as above	38	5.95	43.30	no current reversal				Vapor depos silver, 20% oleic
4½	as above	39	6.3	49.60	no current reversal				Silver spray, 20% oleic
4½	6"dia x 3½"	50(g)	7.5	57.10	no current reversal				Silver spray, 20% oleic
(b)	6"dia x 3½"	37	1.25	58.35	no current reversal				Cleaning sol'n,HCl , 20% oleic
2	6"dia x 3½"	29½	24.45	82.80	15	92	23.6	8	Silver spray, 20% oleic
2	6"dia x 3½"	31½	14.55	97.35	16.1	92	25.3	8	Silver spray, 20% oleic

TABLE 4-1(continued)

Run No.	Remarks
1	Poor adhesion.
2	Fine grained, very ductile deposit, slight peeling at low CD.
3	Excellent deposit.
4	Poor adhesion, excellent deposit.
5	Fine grained deposit, ductile. Coarser at high CD than Run 2; small blisters at solution level.
6	Slight treeing at edges; deposit ductile, rigidly adhering; cannot separate.
7	As above, but coarser deposit. CD varied.
8	Beautiful replication; ductile, separated easily.
9	Good deposit; fine crystalline, blisters at solution level.
10	Easy separation, excellent deposit.
11	Too easily separated, tensile stress noticeable, solution crept under deposit.
12	Stressed (about 600psi at 50asf), but stresses increased only slightly with CD.
13	Stressed, easy separation, laminated when current was interrupted for 12 hrs. Restart in plating cycle.
14	Same as above. Restart in deplating cycle. Spontaneous fine cracks in deposit.

TABLE 4-2

RESULTS OF MATERIALS COMPATIBILITY TESTING
(listing of common materials of construction)

Weighed specimens were placed in closed bottles containing hydride bath for a
duration of 18 days. Specimens extend above liquid level. Weight change and
appearance is recorded.

	ITEM	WEIGHT CHANGE	REMARKS
METALS	Inconel	none	Excellent appearance
	301 stainless steel	none	Excellent appearance
	Carbon steel	-0.02%	Some rusting
	Nickel	none	Excellent appearance
	Silver	none	Excellent appearance
	Copper	none	Excellent appearance
	Brass	none	Excellent appearance
	6061T6 Aluminum	-0.15%	Attacked by liquid & vapor phase
	2024T3 Aluminum	-0.13%	Attacked by liquid & vapor phase
	2S Aluminum	+0.9%	Attacked by liquid & vapor phase
PLASTICS	Teflon	+0.27% (+0.30%)*	Excellent appearance
	Polyethylene	+2.6% (+2.55%)*	Slight discoloration
	PVC	-0.025% (-0.07%)*	Excellent appearance
	Lined fiberglass	-0.12% (-0.18%)*	Excellent appearance
	Tygon	-----	Severe attack by liquid phase
	Synthesine coating (fired 400°F/20min)	-0.11% (-0.21%)*	Excellent appearance
RUBBERS	Neoprene.	-----	Discoloration by liquid phase; minor vapor attack
	Natural rubber	-----	Swelling, discoloration in liquid phase; minor vapor attack
	Viton	-----	Swelling in liquid phase; minor or no vapor attack
MISC	"Plater's tape"	-----	Severe attack by liquid; some by vapor phase; tape acts as wick
	Plain masking tape	-----	Severe attack by liquid; less vapor attack than above; acts more like wick than above
	Thick silver lacquer coating on nickel	-----	Coating intact but lifted off
	Thin silver lacquer coating on nickel	-----	Excellent appearance

* Specimens put back for an additional 18-day exposure. Value in parenthesis re-
presents weight change for total (36-day) exposure.

5. PHYSICAL PROPERTIES OF ELECTROFORMED ALUMINUM

The mechanical and optical properties of the deposited aluminum were measured on approximately 0.030-inch thick electroformed specimens. Milling, machining and grinding of the aluminum deposits to fabricate the test specimens confirmed the integrity of the deposit, since no delamination or other defect resulted from these operations.

5.1 MECHANICAL PROPERTIES

5.1.1 STRESS-STRAIN CURVES

5.1.1.1 TEST SPECIMENS

Four standard tensile specimens (see Fig. 5-3), with a reduced cross-section of 0.406 inch x 0.026 inch, were machined from 0.028 inch electroformed aluminum plate. One specimen was used to determine the proper stress and strain magnification scales. Data from the remaining three are reported below.

5.1.1.2 TEST EQUIPMENT AND PROCEDURE

Stress-strain curves were obtained from tests conducted on a Floor Model TT Instron Testing Machine, using an F-cell for load pick-up. The cell was adjusted to 200 pounds full chart reading. Strain was measured with a Tinius-Olsen S-3 extensometer with a one-inch gauge length, adjusted to produce a 125x magnification. The total elongation was measured over one-inch gauge marks with a vernier calipers. The strain rate of each test was approximately 0.05 inch per inch per minute at the start of the test.

5.1.1.3 TEST RESULTS

Data reduction of the stress-strain curves yielded the following average values: modulus of elasticity = 7.98×10^6 psi; tensile yield strength at 0.2% offset = 7,810 psi; ultimate tensile strength = 11,050 psi; and elongation = 26%. All mechanical property data are tabulated and compared with handbook values of 2S aluminum in Table 5-1. A typical tensile stress vs. elongation curve for electroformed aluminum appears in Figure 5-1. The actual Instron run data has been replotted for one specimen to provide a reproducible copy and appears in Figure 5-2.

5.1.2 THERMAL EXPANSION

5.1.2.1 TEST SPECIMENS

Four $3\frac{1}{4}$" long, $\frac{1}{2}$" wide specimens were machined and surface ground from a 0.028-inch electroformed aluminum plate (see Fig. 5-4). One specimen was used for set-up; therefore, data for the remaining three specimens are reported.

5.1.2.2   TEST EQUIPMENT AND PROCEDURE

The more common dilatometer method of measuring thermal expansion was impractical for the soft, thin, 0.026-inch electroformed aluminum specimens. Therefore, an alternate method was devised. A Model KE-2 Keuffel and Esser Theodolite was mounted at a known distance from the specimen to measure the angle subtended by a 3-inch gauge length of the electroformed aluminum specimen. The change in gauge length was calculated from the measured change in the subtended angle which resulted from the temperature variation. The accuracy of the theodolite is within one-half second of arc.

The test specimens were taped to an aluminum block to prevent buckling. However, the adhesive side of the tape was covered where it could exert any restraint on the specimen. The specimens were placed in a Missimer High-Low Temperature Chamber and sighted during test through a 10" x 10" viewing port.

The selected temperature range of -70°F to $+200^\circ$F was divided into three increments: -70°F to 0°F; 0°F to $+100^\circ$F; and $+100^\circ$F to $+200^\circ$F.

Measurements were made 30 minutes after the specimen temperature had stabilized for each temperature cited above. A shielded thermocouple mounted on the gauge length surface of the specimen monitored the temperature during these tests.

5.1.2.3   TEST RESULTS

An average value of 13.6 x $10^{-6}/^\circ$F over the -70°F to $+200^\circ$F temperature range was measured for the linear coefficient of thermal expansion of electroformed aluminum. These data are tabulated in Table 5-2.

5.1.3   DENSITY

5.1.3.1   TEST SPECIMENS

Two specimens were deemed sufficient because of the routine and simple character of the density determination. Tested tensile specimen "halves" were utilized for this purpose.

5.1.3.2   TEST EQUIPMENT AND PROCEDURES

The density of the electroformed aluminum specimens was determined by the standard ASTM method. All weighings were done with an analytical balance accurate to within 0.1 milligram. The specimen volume was determined by measuring the water displacement weight of the sample.

5.1.3.3 TEST RESULTS

Values of 97.4 and 98.9 percent of theoretical density were measured for the two electroformed aluminum specimens. These data appear in Table 5-2. These low values may be explained by the rough texture of the back of the electrodeposits, which prevented perfect wetting of the deep crevices.

5.2 OPTICAL PROPERTIES

5.2.1 SPECULAR REFLECTIVITY

5.2.1.1 TEST SPECIMENS

Flat aluminum plates, approximately 2 3/8" x 2 3/8" x 0.016" thick, were electroformed over an electroformed nickel tool. Deposition of the aluminum was by our optimized operating conditions which are part of the large GE equipment design. One electroformed aluminum specimen was mechanically polished. This electroform, a second specimen in the as-removed condition, and the nickel master from which these replications were made, were sent to a subcontractor for vacuum coating with aluminum and silicon monoxide, successively.

5.2.1.2 TEST EQUIPMENT AND PROCEDURES

The specular reflectivity of the three optical test specimens was measured at 100^OF over the spectrun of 0.3 to 7.0 microns. A Beckman Model DK1L Reflectometer was used to determine the specular reflectivity in the 0.3 to 2.7 micron spectral range. For the 2.0 to 7.0 micron spectral range, the optical test specimen was measured in a Perkin-Elmer Model 205 Reflectometer. Reflectivity of an aluminum mirror standard was simultaneously determined to enable calculation of the absolute values for each specimen.

5.2.1.3 TEST RESULTS

The specular reflectivity of the electroformed aluminum specimens and nickel mirror appears in Table 5-3. These data indicate that mechanical polishing of the optical surface prior to vacuum coating markedly reduced the specular reflectivity in the 0.3 to 1.5 micron wave length range. The results obtained for the electroformed aluminum closely parallels that of the nickel master.

The subcontractor indicated that the silver film was not a desirable substrate for their coating process, and that, eventually, tarnishing of the silver will show through the reflective coating.

FIGURE 5-1   TENSILE STRESS-ELONGATION CURVE FOR ELECTROFORMED ALUMINUM

FIGURE 5.2: TENSILE LOAD VS. STRAIN ELECTROFORMED ALUMINUM

NOTE:- THIS FIGURE IS A COPY OF THE ACTUAL INSTRON TENSILE TEST DATA
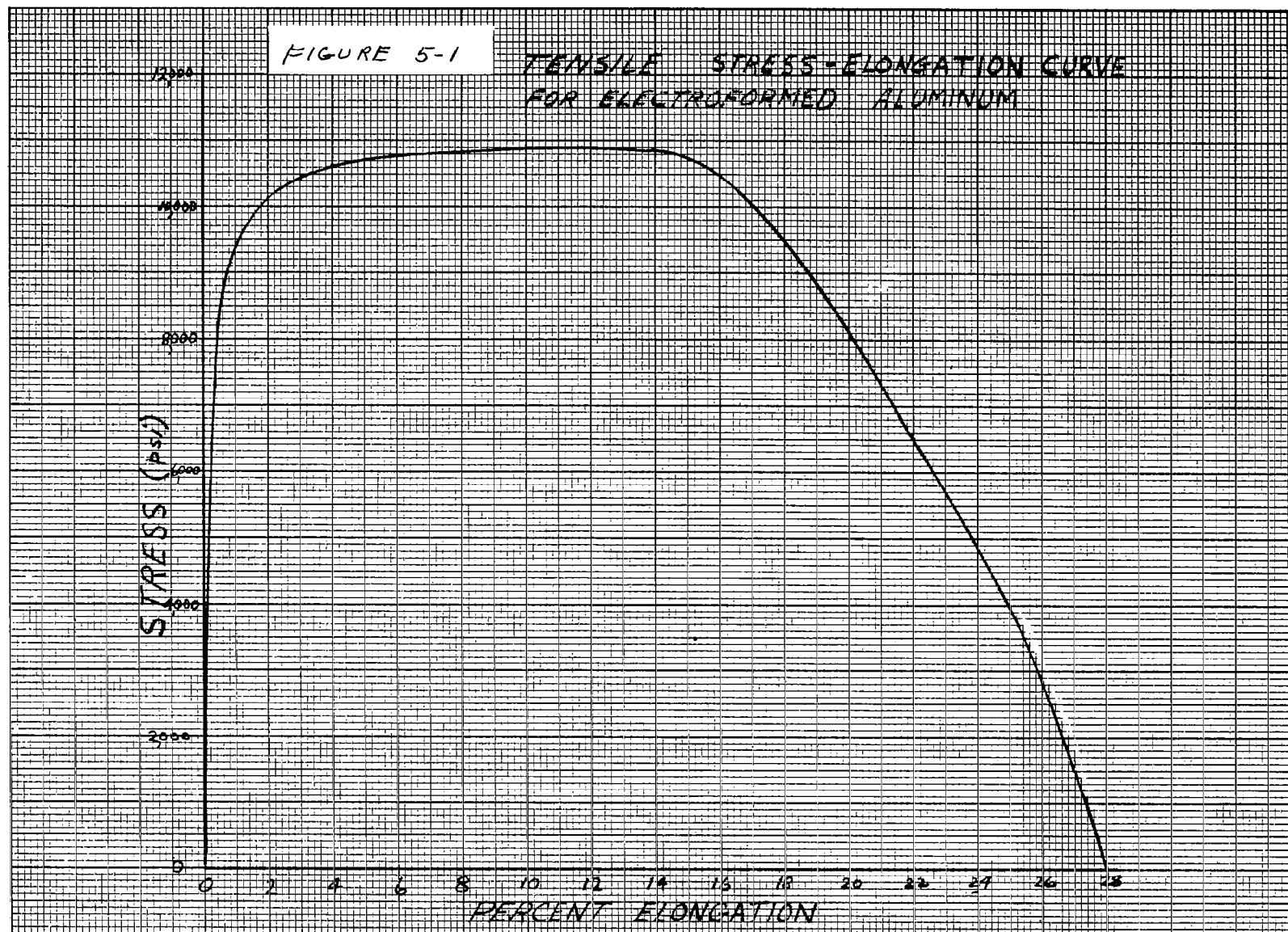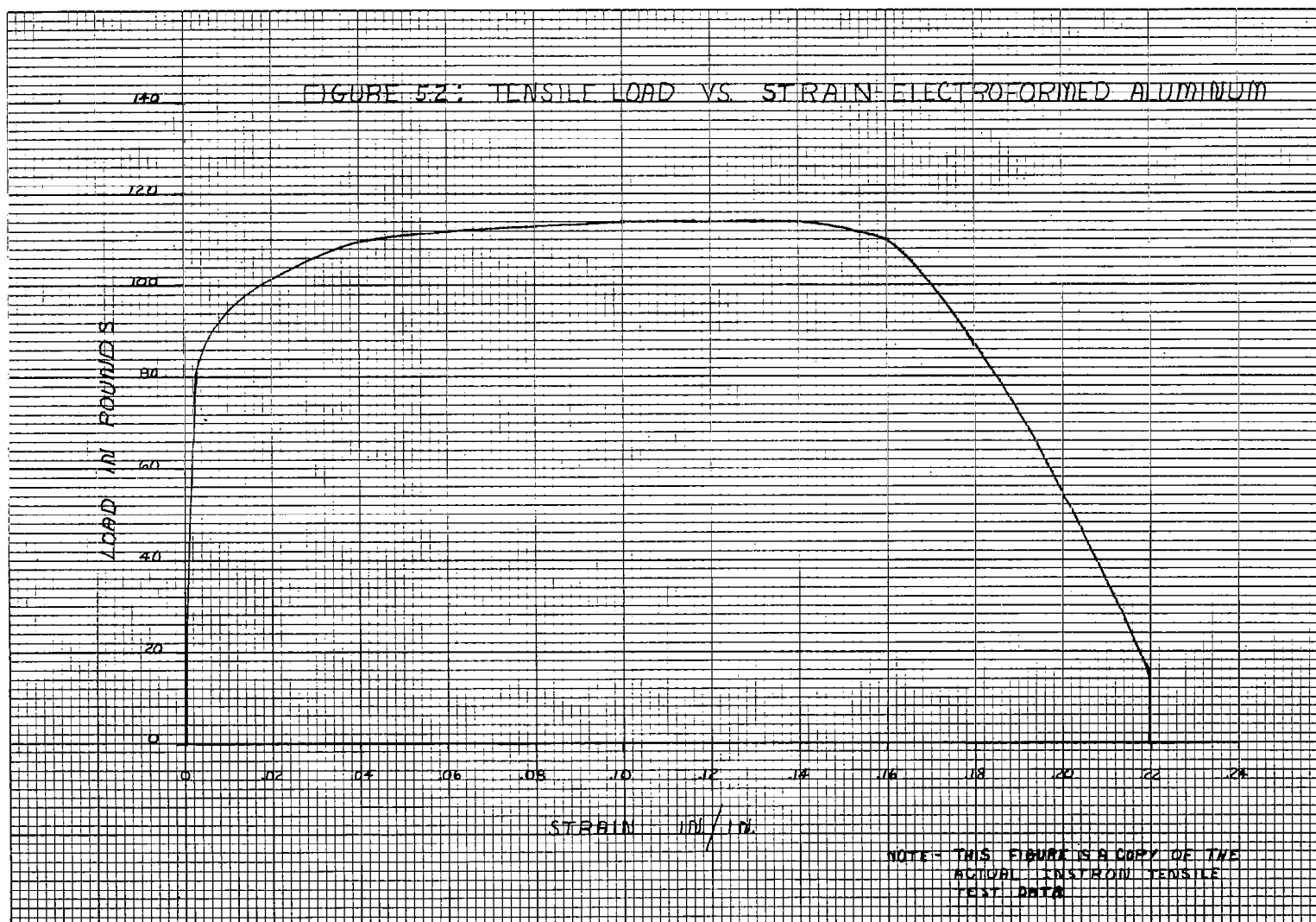
FIGURE 5-3

TENSILE TEST SPECIMENS

Electroformed aluminum tensile test specimens after test.
Specimen No. 4 failed thru radius (separation on right);
the other side was broken by flexing.
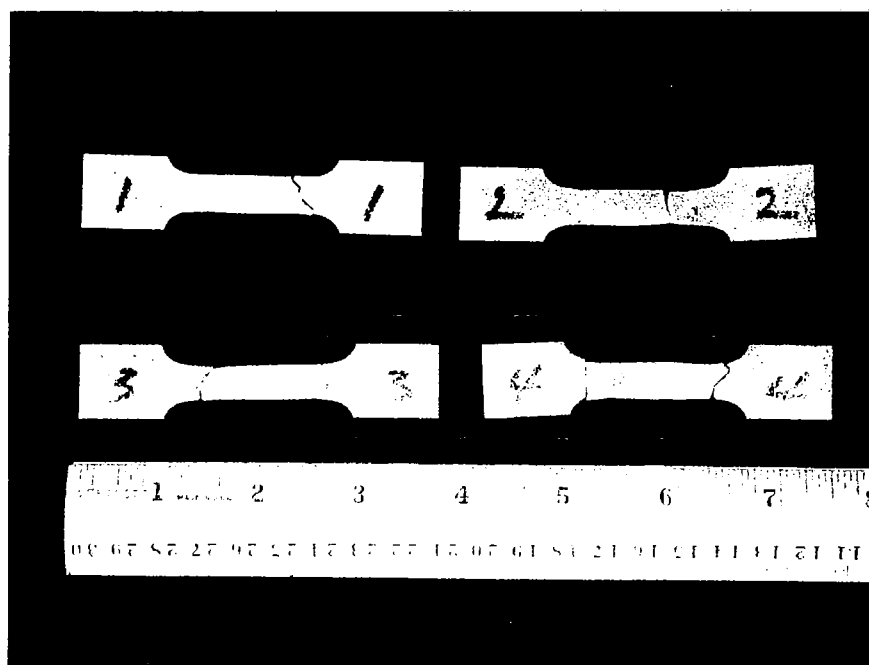
FIGURE 5-4

THERMAL EXPANSION SPECIMENS

Electroformed aluminum linear expansion specimens after test.
Thermal expansion measurement was made with a dilatometer on
the top specimen, but this method proved unsuitable. Accordingly,
linear distance between scribe lines was determined with a
theodolite.
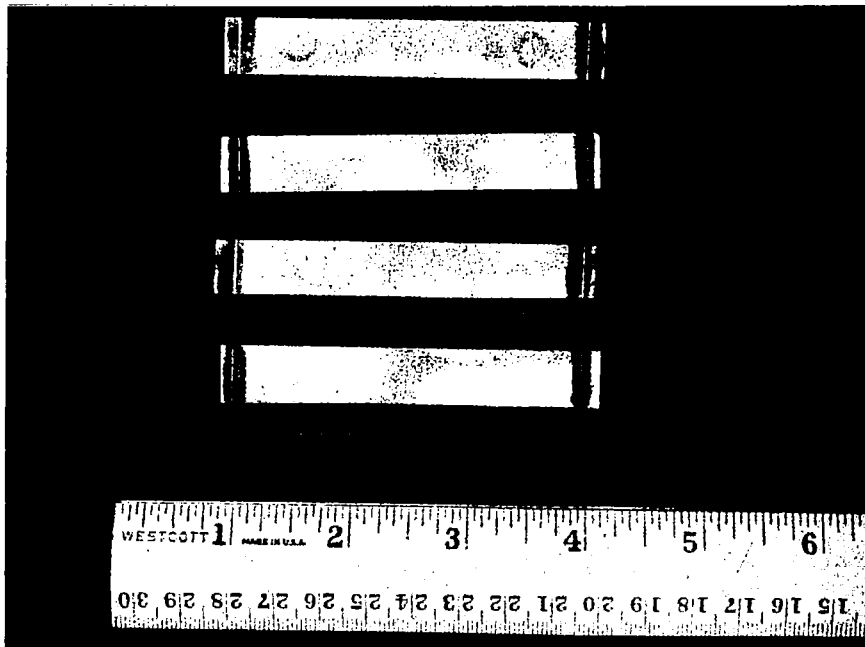
TABLE 5-1

MECHANICAL PROPERTIES OF ELECTROFORMED ALUMINUM

TENSILE PROPERTIES

Specimen Number	Modules of Elasticity (psi)	Ultimate Tensile Str. (psi)	Tensile Yield Str. @0.2% Offset (psi)	Elongation (%)
1	7.83×10^6	10,850	7,880	24
2	8.17×10^6	11,290	(a)	28
3	7.95×10^6	11,000	7,740	(b)
Average	7.98×10^6	11,050	7,810	26
2S Aluminum	10.0×10^6	13,000	5,000	35

(a) Erratic extensometer readings in yield area

(b) Failed through radius area before full elongation was reached

TABLE 5-2

LINEAR COEFFICIENT OF THERMAL EXPANSION

Specimen Number	1st Run	2nd Run	Averages
1	13.2×10^{-6} /°F	14.6×10^{-6} /°F	13.9×10^{-6} /°F
2	13.8	12.9	13.4
3	14.1	12.7	13.4
Average	-	-	13.6
2S Aluminum	-	-	$(12.2 - 13.1)10^{-6}$(a)

DENSITY

Specimen Number			
1	2.64 g/cc (97.4% of theoretical density)		
2	2.68 g/cc (98.9% of theoretical density)		
Average	2.66 g/cc (98.2% of theoretical density)		
2S Aluminum	2.71 g/cc		

(a) Alcoa Aluminum Handbook cites 12.2×10^{-6}/°F from -58° to +68°F and 13.1×10^{-6}/°F from 68° to 212°F.

TABLE 5-3

SPECULAR REFLECTIVITY OF ELECTROFORMED ALUMINUM

All specimens were vacuum coated with aluminum and with silicon monoxide.

Wave Length, microns	Electroformed Aluminum As Deposited, %	Electroformed Aluminum Polished Surface, %	Nickel Master, %
0.3*	83.1	31.4	85.8
0.4	87.3	45.7	87.7
0.5	88.1	56.9	89.0
0.6	88.2	64.2	88.3
0.9	87.2	75.1	88.0
1.2	93.7	86.5	94.1
1.5	95.6	90.9	96.8
1.8	95.8	93.1	96.7
2.1	96.5	94.3	96.7
2.4	96.3	95.6	96.8
2.7	96.0	96.4	97.4
2**	95.3	94.3	96.2
3	95.3	96.0	97.0
4	95.9	95.9	96.6
5	96.2	96.7	97.4
6	95.8	97.2	97.7
7	96.0	96.4	97.8

* Reflectivity determined with Beckman-Model DK1L Reflectometer for spectral range of 0.3 to 2.7 microns.

** Reflectivity determined with Perkin-Elmer Model 205 Reflectometer for spectral range of 2-7 microns.

6. SPINCASTING THE FEMALE EPOXY MOLD

The slow, expensive method of grinding accurate paraboloidal glass masters presents severe limitations where either large diameter surfaces or deep-dish paraboloids are required. In the process of spincasting, the paraboloidal shape is generated solely through the interaction of gravity and centrifugal forces. The basic simplicity of this procedure remains when spincasting a small or large diameter, shallow or deep-dish paraboloid.

A GE proprietary epoxy formulation was tested in the laboratory and found compatible with the electroforming process conditions. A support structure, in which the layers of this epoxy formulation were spincast to produce the female mold, was designed and fabricated. Details are presented below.

6.1 SPINCAST SUPPORT STRUCTURE DESIGN

6.1.1 DESIGN PHILOSOPHY

The structure was designed so that the addition of weight, caused by plating the male nickel master on the epoxy and placing the male back-up structure onto this, would cause a distortion of the female master surface not to exceed one minute of slope. Because of the small relative size of this mirror, the structure was not stress limited in either the metal structure or the epoxy under loadings caused by handling.

Stainless steel was chosen for fabricating the structure to eliminate the need for an elaborate protective coating to protect the structure from the electroforming solution. A simple epoxy coating was used, however, to electrically insulate the structure in the bath and prevent deposition of nickel on the structure.

A single-piece, dish-shaped plate was used for the mold base and supported only at the periphery to prevent any possible show-through from occurring on the epoxy surface due to stiffeners on the mold base.

The tie-down and lift points on the mold were designed so that essentially zero bending load is placed into the dish during handling, and a uniform shear force is the only external load imposed on the dish. Therefore, no distortion of the epoxy mold occurred during handling.

6.1.2 DESCRIPTION OF THE STRUCTURE

The mold support structure was a paraboloidal dish reinforced by a ring assembly, which was welded to the outer periphery of the dish. This structure is shown in Figure 6-1.

The dust cover was formed by a vacuum-temperature process, using the mold as a die, thereby insuring uniformity of the cover contour with the dish surface.

A 28" spherical radius dish was used to approximate the paraboloidal shape of the final spincasting. This radius so closely approximated the paraboloidal shape that a uniform thickness of epoxy was formed over the usable area of the spincast surface.

Because an air pocket would be formed when the structure was lowered into the electroforming bath, holes were drilled into the cylindrical shell, which formed the web of the reinforcing ring, near the top of the ring assembly to permit air escapement.

The entire support structure was stress relieved prior to spincasting.

6.2 PREPARING FOR SPINCASTING

6.2.1 SPINCAST FACILITY

A small development spincasting facility was used for this program. The spincasting table is shown in Figure 6-2. This facility can accommodate molds up to 7 feet in diameter and up to 1500 pounds in weight. The operating speed range is from 10rpm to 125rpm. Although this table has neither vibration damping nor an accurate speed mechanism, it has been used to obtain quality spincastings.

6.2.2 ALIGNMENT OF SPIN TABLE

The spin table was checked with a "precision machinists" level (0.005" per foot gradations), placed at the outer periphery of the 5-foot diameter table top. Precision dial indicators were mounted against stationary parts of the spincast table to measure wiggle during rotation. None was noted.

6.2.3 CALCULATION OF THE ROTATIONAL SPEED

The focal length of the paraboloid and the rotational speed during spincasting are related by the following equation:

$$f = \frac{450g}{\pi^2 N^2}$$

where f = focal length, in feet; N = rotations per minute; g = gravitational constant, ft/sec^2.

The desired focal length was 13.0 inches. The gravitational constant was measured at Franklin Institute, Philadelphia, Pa. (about 6 miles from the spincasting facility) as 32.1587 feet/sec/sec.

$$N = \frac{1}{\pi}\sqrt{\frac{450g}{f}} = \frac{1}{3.14159}\sqrt{\frac{450 \times 32.1587 \times 12}{13.0}}$$

$$N = 36.795 \text{ or } 36.8$$

6-2

6.2.4 ROTATIONAL SPEED MEASUREMENT, CONTROL AND ADJUSTMENT

The rotational speed for spincasting the epoxy mold (36.8rpm) was monitored on a
continuous basis, by measuring the period of revolution using an Eput meter with
an optical sensing head. During the entire process, the change in rotational
speed was less than 0.4%, and no corrective adjustments were required.

Adjustment of the rotational speed, if it had been necessary, would have been by
manually adjusting the diameter of a V-belt pully drive.

6.2.5 PRETREATMENT OF SUPPORT STRUCTURE

The epoxy mold support structure surface, upon which the epoxy rests, was pre-
treated to effect increased adhesion and provide a flexible buffer layer to mini-
mize the effect of the differential shrinkage between the stainless steel and the
epoxy layers. To accomplish this, the surface was roughened by grit-blasting,
then cleaned with air and degreased thoroughly. A thin, flexible layer consisting
of Araldite 571KX and Pentamide 815 thinned with toluene was painted on. This
was then oven-cured at 130^OF for a minimum of 8 hours. The results of deviation
from this process are reported in section 6.3.

6.2.6 MIXING AND METERING OF THE EPOXY FORMULATION

Machine mixing vs. hand mixing, for the small quantity of epoxy required for spin-
casting each layer, was evaluated. Hand mixing proved superior and was employed
in all successive pours.

After hand mixing this proprietary epoxy formulation, the material was outgassed
in a 5-gallon vacuum chamber, with a transparent top to allow visual monitoring of
this step. A pressure of 2mmHg absolute was maintained in the chamber. The epoxy
was funnel-poured into the rotating support structure. The cover was maintained
on the curved surface throughout the process to prevent dust settling on the epoxy
surface. Cleanliness during the entire process is essential.

Static pours were made into pie plates to secure process evaluation samples.

6.2.7 THERMAL CURE

The spincast was rotated several days until an ambient temperature set had occurred.
The epoxy mold was subsequently removed from the spin table and oven-cured. A slow
heat-up and cool-down cycle was employed to prevent thermal shocking the mold.
The cure temperature was 120^OF for about 12 hours.

6.3 FIRST SPINCAST SERIES

The support structure for the epoxy mold was mounted on the spincasting table,
which was then balanced to insure a ripple-free surface and the specified geometry.
Figure 6-2 shows the spincast mold in position on the spincasting table. The epoxy
spincast mold is shown in Figure 6-3.

After room temperature cure of the second spincast epoxy layer was completed, the
surface was inspected visually. The geometry and surface finish were excellent;
and after some deliberation, it was decided to eliminate the scheduled third pouring.
Subsequently, the epoxy spincast geometry was not within requirements, which were
less than 2 minutes average angular error over 95% of the area. Therefore, it was
decided to spincast a new epoxy mold.

Removal of the epoxy layers from the support structure revealed that the primer,
which was applied to the support structure prior to spincasting, was loosely ad-
hering to or separated from the metal support structure, causing the slope errors
noted on the spincast surface. This separation, in all probability, occurred during
the cure cycle, which followed the visual inspection. Critical analysis of the
primer application and cure procedures revealed that some deviation from the stan-
dard method had occurred, due in part to the smaller size of the spincasting table
presently used. In spincasting the second epoxy mold, the techniques developed and
used on the previous 9½-foot mirror were rigidly adhered to.

6.4 SECOND SPINCASTING SERIES

After the room temperature cure of the second spincast epoxy layer was completed,
the surface was inspected visually. An excessive number of bubbles on the epoxy
surface made the female mold unsatisfactory for electroforming the male nickel
master. A third spincast epoxy layer yielded a similarly unsuitable surface. The
cause of the excessive number of bubbles was traced to "aging" of the initial epoxy
resin mixture, which occurred while the first spincast series was thermally cured
and inspected.

The epoxy layers were removed from the metal support structure. A fresh batch of
material was prepared to spincast the third epoxy mold. The procedures previously
established were again rigidly adhered to.

6.5 THIRD SPINCASTING SERIES

After the room temperature cure of the second spincast epoxy layer was completed,
the surface was inspected visually. An excellent specular surface was noted,
marred only by 8 small-sized bubbles on the outer edge. The affected area was
less than 0.5% of the total surface. The mold was optically inspected before re-
moving from the spincast table for thermal curing. All measured slope errors were
10 seconds or less.

After thermal cure, the epoxy mold was optically inspected again. The data, presented in Table 6-1, indicate the female mold had an average angular slope error of 1 minute 17 seconds (1.29 minutes). No measured region exceeded 2 minutes. Figure 6-4 indicates the inspection locations.

Since the epoxy mold geometry met the contractual requirements, and the surface was of such high specular quality, the spincast was deemed satisfactory despite the 8 bubbles previously mentioned. However, to prevent possible difficulties during separation of the electroform, the bubbles were patched with an SiO_2-filled epoxy resin.

6.6   FOURTH SPINCASTING SERIES

Difficulties in electroforming the male nickel master, described in the male master electroforming section, necessitated spincasting a new female epoxy mold. The old epoxy layers were removed from the metal support structure by thermal shocking. A fresh batch of material was prepared to spincast the new mold. The procedures, previously used, were again rigidly adhered to.

After a room temperature cure of the second spincast epoxy layer was completed, the surface was inspected visually. The surface specularity was marred by 4 small-sized bubbles on the outer edge and about fifteen 1/8 to 1/4-inch diameter shallow areas originating from broken bubbles. Since the total affected area was less than 0.5% of the surface, the mold was thermally cured, and subsequently optically inspected. The data, presented in Table 6-2, indicate that the female mold had an average angular slope error of 0.66 minutes, and that no measured region exceeded 1.54 minutes. To prevent difficulties during separation of the electroform, the four bubbles were ground off, and the voids were filled in with an epoxy resin.

6.7   OPTICAL INSPECTION OF SPINCAST EPOXY MOLD

The slope errors were measured on the spincast epoxy mold at the locations shown in Figure 6-4. Adjustments were made to achieve the "best fit" focal point location of the light source. The adjusted slope errors are presented in Tables 6-1 and 6-2.

6.7.1   PROCEDURE

Two theodolites were mounted over the spin table. The outer and inner scribe lines on the epoxy were sighted while the table was rotated to determine whether the mold was centered. The mold was adjusted until the radial variation was within the measured variation in scribe line width.

The focal point was found by locating a wheat grain light source, such that the lamp filament could be observed in the theodolites at all radial inspection locations. This represents the "best fit" condition.

The inspection was accomplished by leveling the theodolite over the radial inspection stations, and aligning the telescope to bring the lamp filament in the center of the reticle. The angle of inclination of the vertical and the azimuth were recorded. The spin table was then rotated to inspect each of 12 angular stations.

6.7.2   CALCULATION

Test measurements were divided by two to obtain slope error, since the measured angle of inclination is two times the slope error. The error in the lateral location of the light source at the focal point was eliminated by adjusting the data, as described in Ref. 15.

SECTION **A-A**

Figure 6-1. Spincasting Mold

2.00 TYP
.400 TYP

A

120° TYP

33.00 DIA

A

SPINCASTING MOLD

FIGURE 6-1

FIGURE 6-2

SUPPORT STRUCTURE FOR THE SPINCAST MOLD

(shown mounted on the 5' spincasting table)
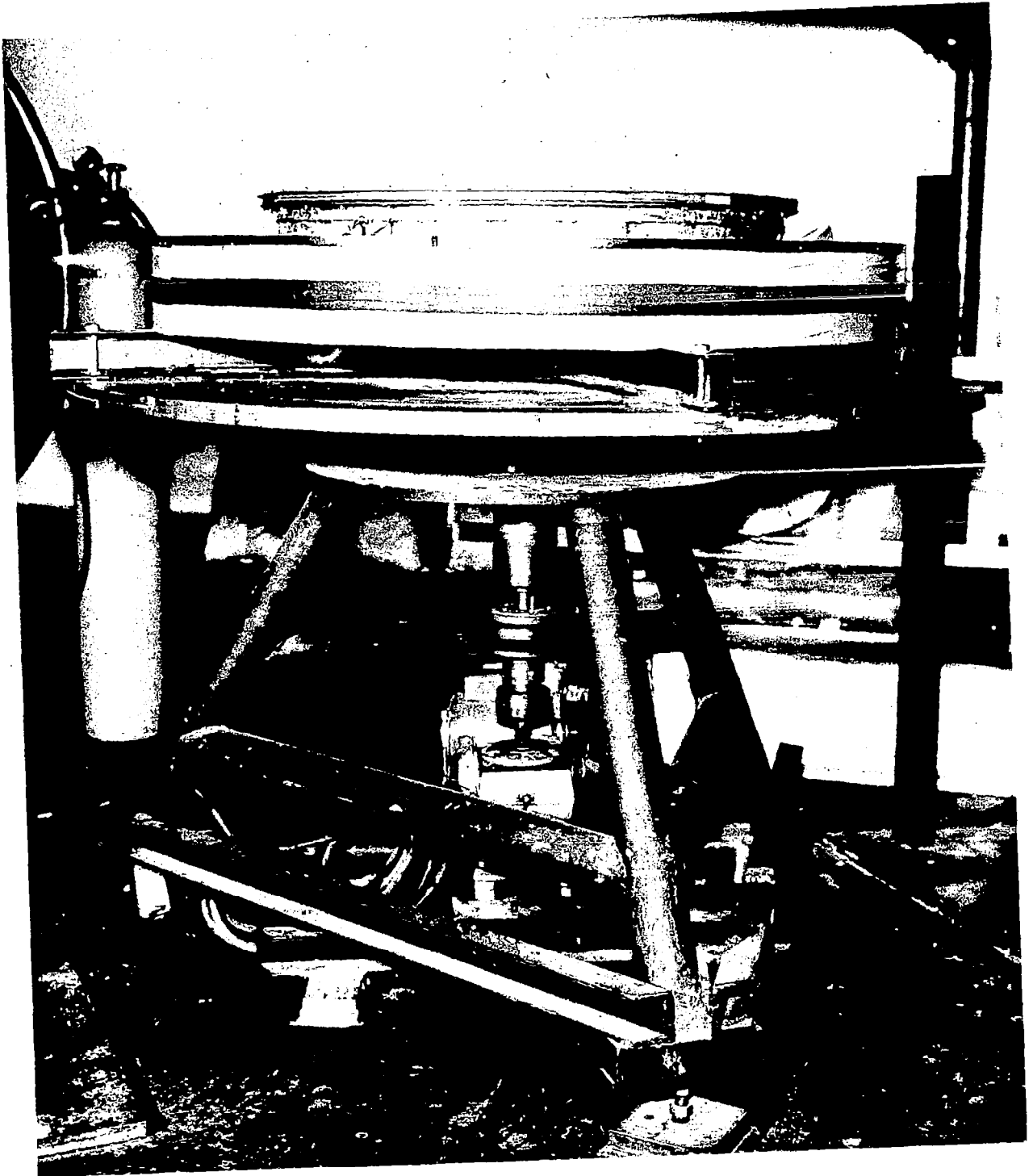
FIGURE 6-3

CLOSE-UP VIEW OF FEMALE EPOXY MOLD

(Epoxy spincast is firmly bonded to stainless steel support structure)
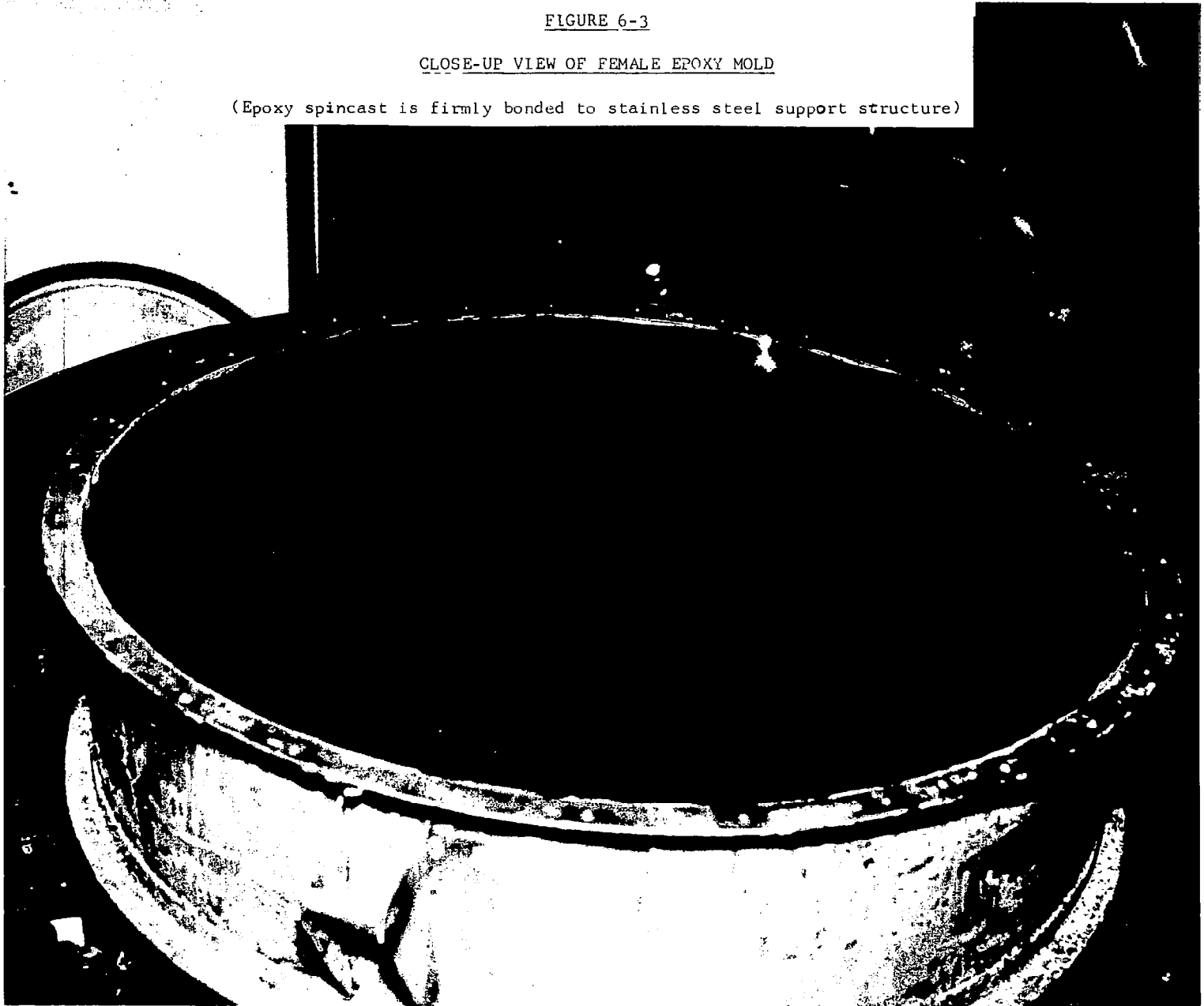
FIGURE 6-4

INSPECTION LOCATIONS

PICK-UP POINT

A-1

A-2 (ANGULAR STATION)

C-4

RADIAL INSPECTION LOCATION

ALIGNMENT PIN (3 PLACES)

C-3

5

A-3

4

3

C-2

A-4

OUTER SCRIBE LINE

C-1

B-1

30° (TYPICAL)

MOLD SUPPORT CHANNEL
(NO PIN THIS EAR ONLY)

B-4

B-3

B-2

3/4"
3"
3"
3"
3"
3"

5
4
3
2
1
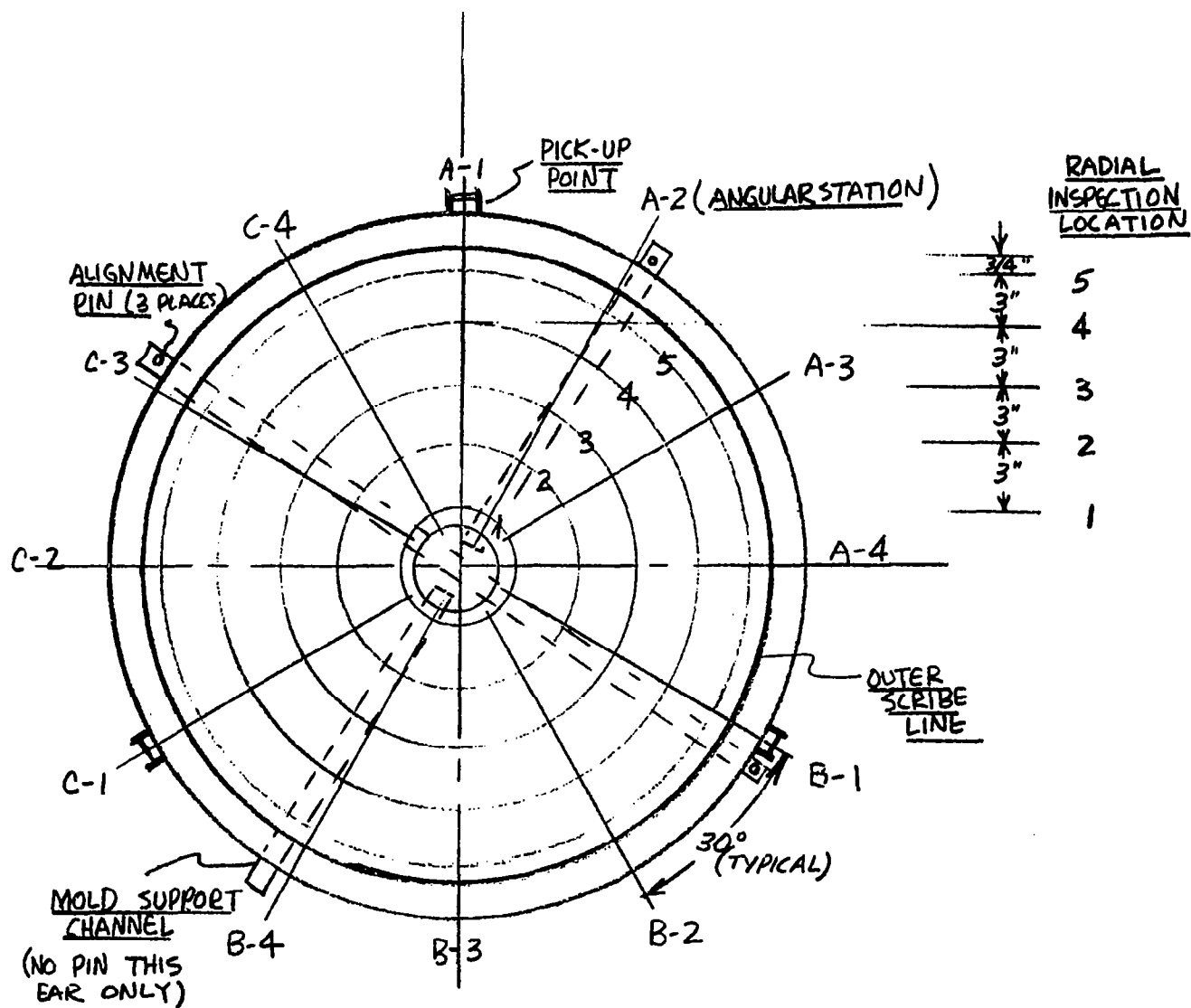
TABLE 6-1

ADJUSTED SLOPE ERRORS (MINUTES)

for 30" epoxy spincast   focal point: 13 ± 1/16 inch

THIRD SPINCASTING SERIES

Angular Station*	Inspection Diameter (inch)				Average
	10	16	22	27-5/8	
A-1	1.75	1.38	0.40	1.38	1.23
A-2	1.50	1.44	0.96	1.48	1.34
A-3	1.52	1.64	1.22	1.01	1.35
A-4	1.73	2.00	1.44	1.00	1.54
B-1	1.61	1.95	1.37	0.62	1.39
B-2	1.53	1.64	1.53	0.40	1.27
B-3	1.27	1.38	1.34	0.20	1.00
B-4	1.08	1.25	1.25	0.50	1.02
C-1	1.38	1.16	1.30	0.81	1.16
C-2	1.41	1.00	1.59	1.61	1.40
C-3	1.54	1.28	1.69	1.46	1.49
C-4	1.67	1.38	0.74	1.31	1.28
Average	1.50	1.46	1.24	0.98	1.29

*Refer to Figure 6-4

TABLE 6-2

ADJUSTED SLOPE ERRORS (MINUTES)

for 30" epoxy spincast   focal point: 13 ± 1/16 inch

FOURTH SPINCASTING SERIES

Angular Station*	Diameter Location (inch)				
	10	16	22	28	Average
A-1	0.77	0.70	0.95	0.52	0.73
A-2	0.52	0.22	1.17	1.22	0.80
A-3	0.82	0.18	0.58	1.17	0.68
A-4	0.75	0.18	0.27	0.62	0.45
B-1	0.72	0.20	0.27	0.43	0.40
B-2	0.57	0.23	0.20	0.47	0.37
B-3	0.40	0.30	0.32	0.52	0.38
B-4	0.22	0.28	0.67	1.24	0.60
C-1	0.52	0.52	1.27	1.49	0.95
C-2	0.85	0.73	1.54	1.40	1.13
C-3	1.07	1.05	0.70	0.87	0.92
C-4	0.95	0.50	0.10	0.42	0.49
Average	0.68	0.42	0.67	0.86	0.66

*Refer to Figure 6-4

7. ELECTROFORMING THE MALE NICKEL MASTER

7.1 VENDOR SELECTION

A work statement for electroforming the nickel male master (Appendix A) was
sent to several vendors. Vendor quotations for electroforming the male nickel
master were evaluated. Bart Manufacturing Corporation was selected on the basis
of their low bid and prior electroforming experience.

7.2 PROCESS

The epoxy mold surface was cleaned with a mild alkaline cleaner (20 g/l $Na_2 CO_3$,
10g/l $Na_2 HPO_4$, 2ml/l Triton X-100), rinsed with distilled water, sensitized with
a freshly prepared $SnCl_2$ solution (20g/l $SnCl_2$ + 10ml/l HCl), rinsed with distilled
water and silvered by chemically reducing an ammoniacal silver solution.

The silvered epoxy mold was then lowered into the nickel sulfamate bath and approxi-
mately 0.0015-inch of nickel was deposited on the mold surface, followed by a 0.001-
inch copper flash coating. This procedure permitted the "grow-in" of a stiffening
ring (described in section 7.6.2) into which was subsequently inserted a 3-legged
member (spider) for mounting into the aluminum electroforming bath.

The assembly was then returned to the nickel sulfamate solution for additional de-
position to attain a 1/4" to 3/8" thickness. A rotating conforming anode was used
to provide uniform current distribution.

Periodic stress checks in GE's Stress Cell by RSD personnel permitted the selection
of the optimum process conditions for electroforming the male nickel master. RSD-
installed instrumentation enabled rigid process control.

The nickel sulfamate bath composition and process conditions used are listed in
Table 7-1.

7.3 ELECTROFORMING DIFFICULTIES IN THE FIRST ATTEMPT

After the first week of the three weeks' scheduled deposition, an inspection of the
deposited surface revealed the presence of numerous uniformly distributed holes
about 1/8" deep (or, to within 30-40 mils of the initial flash deposit). "Show-
thru" distortion of the optical surface was expected and, therefore, the run was
terminated.

The electroform was separated from the mold, and subsequent inspection revealed
that the decision was right, the electroform would have been a borderline case.
The pits on the back surface showed through faintly as depressions - distortions.

It was established that the holes were evidently caused by hydrogen bubble pitting, i.e. gas retained at the surface blocked further deposition, and subsequently, holes developed at each site. This should not have occurred, and it was believed that the vendor's vacation schedule (shutdown of their plant) somewhat relaxed the agreed-upon process controls.

7.4   CORRECTIVE PROCEDURES

To overcome the difficulties described above, the concentration of wetting agent in the nickel sulfamate plating solution was increased, and an auxiliary circulation system to the mold was added. Deposition on a prototype geometry test specimen proved the effectiveness of the corrective measures.

7.5   ELECTROFORMING THE SECOND MALE NICKEL MASTER

The newly spincast female mold was processed, as previously described. After 8 days residence in the bath the male nickel master was inspected without interruption of the process and freedom from pitting, and a relatively smooth surface was found. The deposition continued for a total of 20 days.

Separation from the epoxy substrate proceeded smoothly by thermal shocking with hot and cold water. Although the epoxy cracked during this separation, the epoxy surface was still highly glossy, unaffected by the electroforming cycle. This was a marked improvement over past years' experiences, when some attack of the epoxy surface invariably occurred, either during the preparatory cycle or during the first few minutes in the plating solution. The significance of this observation is that it shows the soundness of the presently used epoxy pre-plating scheme, and also indicates that, with some improvement of the separation technique, the spincast tool could become reusable.

7.6   MALE MASTER SUPPORT STRUCTURE

7.6.1   DESIGN

It was the purpose of the male master support structure to maintain the paraboloidal shape of the ¼" thick nickel master during separation, electroforming of the female mirror, and final shipment to the customer. Two designs were considered; one was an all-metal structure which was attached to the nickel electroform by electroforming during the process of making the master; the other consisted of a glass reinforced plastic structure which would be foam bonded to the electroformed nickel skin. The all-metal structure was chosen because the aluminum electroforming solution attacked all of the plastics considered.

The support structure is described in Figures 7-1a, b and c. This structure consists of two major parts, a circular ring, and a three-leg spider member which is attached to the inside of the ring. To this spider is attached a stub shaft which is required for rotating the master in the aluminum electroforming bath. Because the spider would interfere with the electroforming process of the nickel master,

the spider was fabricated as a separate unit and attached to the ring support at the completion of electroforming, and prior to separation of the master from the epoxy mold. The three places where the spider is attached to the support ring were prefitted during fabrication of the ring and spider, so that no stresses were introduced into the ring when the spider was assembled, and to eliminate the need for shimming at assembly.

The entire spider assembly was fabricated from stainless steel to eliminate the need for any type of protective coating either in or out of the aluminum electro-forming bath. To permit attachment of the ring to the electroform by electro-forming, it was necessary that the point of the ring be fabricated from nickel. It was also necessary that this point be extremely sharp along the edge to insure sealing in this area between the ring edge and epoxy mold surface. To aid in the ease of manufacturing and reduce the cost of this ring, a solid ring of nickel was not used. The ring was fabricated from hot rolled steel and nickel plated after machining. However, because a final grinding and polishing was required on the point edge of the ring, a build-up of nickel weld material was used in this area.

Holes in the outer edge of the fing were also provided for attachment of the anode guide ring (shown in Figure 7-1c).

The assembly was stress relieved prior to usage to eliminate any possibility of distortion taking place during aging or handling.

7.6.2   ATTACHING THE MALE MASTER SUPPORT STRUCTURE

A thin layer of nickel (approximately 0.0015-inch) was deposited on the female epoxy mold under process conditions described earlier. The nickel deposit was subsequently followed by a 0.001-inch copper flash coating. Separately, the knife edge portion of the support structure (see Fig. 7-1a) was covered with a like cop-per flash coating. The copper coated knife edge was positioned and clamped down on the copper-flashed nickel layer, and the entire assembly was returned to the nickel sulfamate bath for the prescribed 3 weeks of deposition.

It was necessary to resort to this procedure to obtain a sound grow-in of the sup-port structure. A nickel-to-nickel bond would tend to delaminate and be a source of difficulty during the remainder of the program.
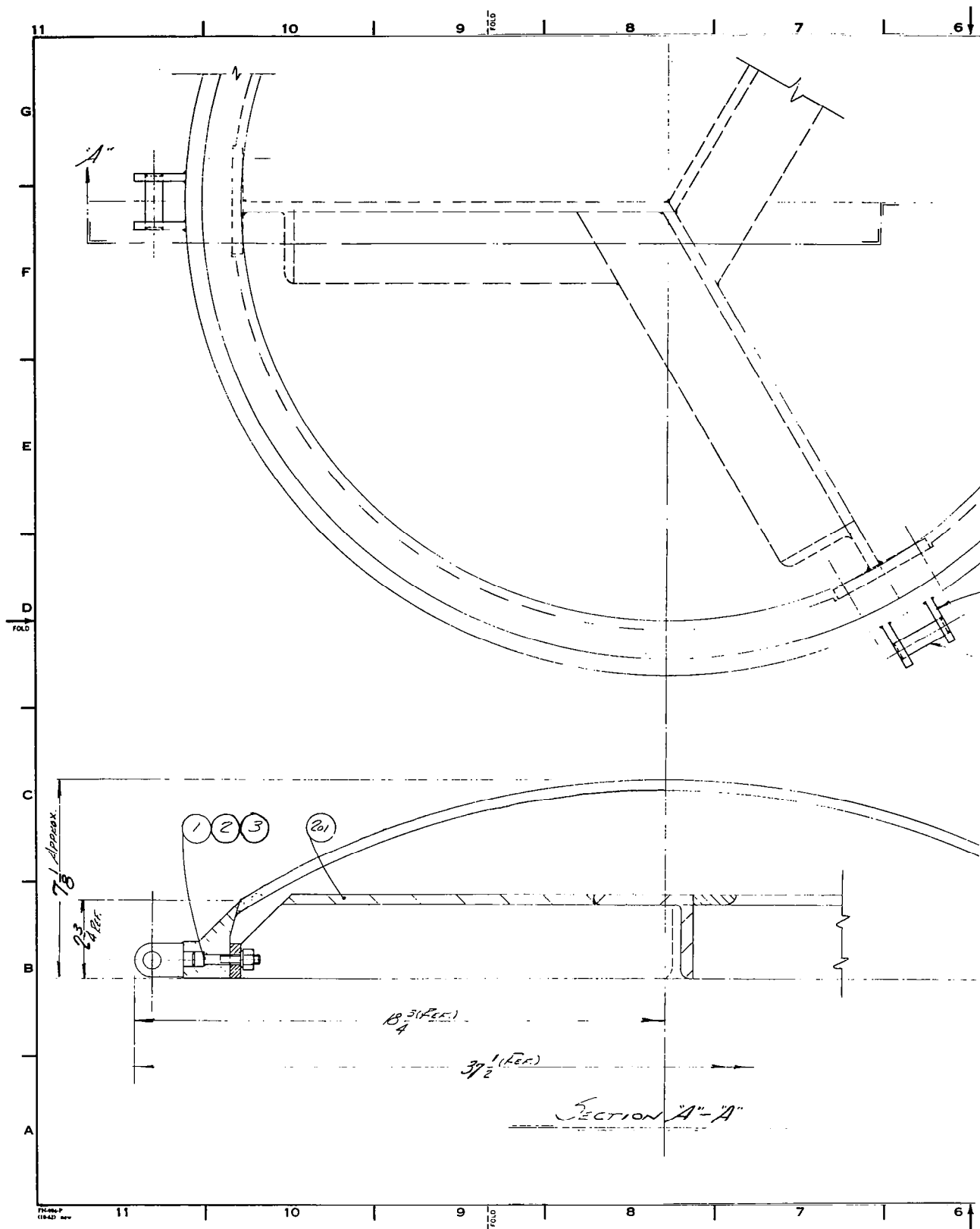
11   10   9 FOLD   8   7   6

G

"A"

F

E

D
FOLD

C

① ② ③   ⑳₁

$7\frac{1}{8}$ Approx.

$2\frac{3}{4}$ B.R.

B

$18\frac{3}{4}(REF.)$

$39\frac{1}{2}(REF.)$

A

Section "A"-"A"

11   10   9 FOLD   8   7   6

7-4

FIGURE 7-1a

REVISIONS

SYM	ZONE	DESCRIPTION	DATE	APPROVED
1				

NOTE:
FOR ALL DETAILS SEE SHEETS
2 & 3

STAMP TOTAL WEIGHT
OF ASS'Y

ITEM	QTY	MAT'L	ROUGH SIZE	DESCRIPTION
3	6	STAINLESS STL	3/8-16 THREAD	HEX NUT
2	6	STAINLESS STEEL	STD 3/8	LOCK WASHER
1	6	STAINLESS STL	3/8-16 x 2 LG	SOC. HEAD SCREW

BILL OF MATERIAL

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES—
TOLERANCES ON:
2-PLACE DECIMALS ±
3-PLACE DECIMALS ±
ANGLES ±
ALL SURFACES ✓
MATERIAL –
GOVT OR COML.
GE

SIGNATURES	DATE
DRAWN	
CHECKED	
ISSUED	

GENERAL ⊕ ELECTRIC
SPACECRAFT DEPT. LOC. VALLEY FORGE

TITLE
NICKLE MAKE MASTER AND
SUPPORT STRUCTURE FOR
30" MIRROR

CONTRACT NO.

CODE IDENT NO.	SIZE	DWG NO.
	D	JK5-0250

SCALE 1/2 SIZE   WT   SHEET 1 OF 3

		REVISIONS		
SYM	ZONE	DESCRIPTION	DATE	APPROVED
A		(1) _Revise Tbl 203 : Increase length._ _C Smith_	4/60	

G

F

E

D
FOLD

BILL OF MATERIAL

ITEM	QTY	MATERIAL	ROUGH SIZE	DESCRIPTION
205	1	STRUCT. ANGLE	3 × 3 × 3/8 × 15 1/2 LG	ANGLE
204	1	STRUCT. ANGLE	3 × 3 × 3/8 × 16 5/8 LG	ANGLE
A 203	3	STAINLESS STL (304)	1/2 × 1 1/4 × 4	MOUNTING PLATE
202	1	STRUCT. ANGLE	3 × 3 × 3/8 × 16 5/8 LG	ANGLE
201	1	STAINLESS STL (304)	WELDMENT OF ITM 202 THRU 205	SUPPORT STRUCTURE

GENERAL NOTES:
IDENTIFY TOOL AS SHOWN.
BREAK ALL UNNECESSARY SHARP
EDGES.
NORMALIZE BEFORE FINISH MACHINING

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES—
TOLERANCES ON: FRACTIONS
2-PLACE DECIMALS ± 5. 1/32
3-PLACE DECIMALS ±
ANGLES ± 0° 30'
ALL SURFACES ✓
MATERIAL—
GOVT OR CONL.
GE

SIGNATURES / DATE

GENERAL ⊕ ELECTRIC
SPACECRAFT — DEPT LOC VALLEY FORGE

TITLE
SUPPORT STRUCTURE
FOR NICKLE MALE MASTER
30" MIRROR

CONTRACT NO.

CODE IDENT NO.	SIZE	DWG NO
	D	SKS-0250

SCALE 1/2 SIZE   WT CALC ACTUAL   SHEET 2 OF 3

FIGURE 7-1b

"B"

18

15°
TYP.

5 DIA
8

2 TYP.

4 TYP.

15°
TYP.

30°
TYP.

15 3/8 S.F. FOR IT. #201
MATCH MARK
LOCATIONS

9/32 DIA THRU
TYP (6) HOLES

3 TYP
8

15°
TYP.

303

301

302

ALL WELDS 1/8 FILLET
ALL AROUND

305 304

TOP VIEW LESS IT. # 306

ELECTRO-FORMED
PORTION (REF)

5 C.
BORE

5 R.

5 DIA.
8

DRILL & C. BORE FOR 3/8-16
SOC. HD. SCR. - TYP (3)
PLACES - LOC. FROM IT. #301
AT ASS'Y - TOTAL OF (6)
HOLES

SECTION "B-B"

7-8

FIGURE 7-1c

TABLE 7-1

COMPOSITION OF NICKEL SULFAMATE BATH

Metallic nickel	6.9 oz/gal
Nickel Sulfamate	29.6 oz/gal
Chloride content	1.2 oz/gal
Boric Acid	4.0 oz/gal

PROCESS CONDITIONS

pH	4.5-4.6
Surface tension	32.5-33.4 dynes/cm
Agitation	By impinging solution
Filtration	Continuous
Electrolyte purification	Continuous
Temperature	$120^{\circ} \pm 0.5^{\circ}F$
Current density	Approx 20 asf (specific values as indicated by GE's periodic stress checks)

8. DESIGN AND FABRICATION OF THE 30" MIRROR ELECTROFORMING EQUIPMENT

A flow diagram, illustrating all equipment and process line requirements for electroforming the 30" diameter mirrors, was prepared. This was based on laboratory results and considered all available information (experimentally rechecked, where doubtful) in the published literature. A work statement, encompassing the flow diagram, was prepared (see Appendix B) and sent to several vendors for quotation.

8.1   REQUIREMENTS FROM THE VENDOR

The vendor's responsibility was defined as the detailed design, fabrication and installation (at a site provided by the vendor) of the aluminum electroforming pilot plant equipment and appurtenances, under GE's direction. Manpower was to be supplied by the vendor for the duration of the aluminum electroforming operation. This manpower would operate directly under GE/RSD for the duration of the program.

8.2   SELECTED VENDOR

Bart Manufacturing Corporation, Newark, New Jersey, and General Technologies Corporation, Alexandria, Virginia, submitted formal bids; Bart was the low bidder.

Bart Manufacturing has considerable experience in general electroforming, a good engineering department, their own equipment fabrication facility, and a considerable pool of useful equipment from which they could draw, as necessary. Since Bart would execute RSD's design and work under RSD's direction, their lack of direct aluminum electroforming experience was not objectionable. Therefore, Bart Manufacturing was selected on a technical and cost basis.

8.3   FINAL DESIGN

The initial flow diagram, as described in the work statement, was modified slightly and is shown with greater detail in Figure 8-1, illustrating the piping and electrical lines and equipment placement for our design. Assembly drawings for the plating tank with its glove box, solution mixing tank with its glove box, and filtration unit appear as Figures 8-2, 8-3 and 8-4, respectively.

8.4   EQUIPMENT FABRICATION AND ASSEMBLY

The plating and storage tanks were fabricated from nickel-clad steel. Cooling coils were subsequently connected to each tank. The plating tank was then teflon-lined to prevent dipole effect during electrodeposition. The stainless steel filters and the two glove boxes (one for each tank) were received from vendors. An air-driven (explosion-proof) crane was installed in a building constructed to house the electroforming equipment. At this point, assembly of the equipment was ready to begin.

Each piece of equipment was positioned, as indicated in Figure 8-1, and leveled. All connecting lines, which would handle liquid were 316 stainless steel, as were all control (ball) valves. The inert gas purge and vent line systems (copper tubing with brass fittings) were concurrently installed.

After completion of the equipment assembly, the checkout procedures revealed numerous small leaks at various joints, etc. Stoppage of these leaks proved a lengthy (10 working days) and frustrating task, with the many threaded stainless joints and piping, ports, covers, etc. Many of the joints had to be welded when all other measures failed, since any leakage would constitute a serious safety hazard because of the explosive and toxic nature of the vapor.

After all leaks were repaired and the final checkout was completed, provisions were made for solution preparation and subsequent electroforming of the two 30" diameter mirrors.

2-3 PSI
N_2 AR

2-3 PSI
N_2 AR

15-25 PSI
N_2

(3) VALVES

BUBBLER

VALVE $\frac{1}{4}$

PRE-CONTACT (D.C.)

GLOVE BOX

TO VENT

RELIEF VALVE (3 PSI)

CONDENSER

$\frac{1}{4}$ VALVES

VALVE

SWIT FLO. WHE

OSCILLOGRAPH

RUPTURE DISC

(2) VALVES

PLATING TANK

QUICK COUPLING

(3) VALVES

PLATING CELL

AGIT. CYL.

PERIODIC REVERSE

MOLD

(2) VALVES

DIODES

ANODE

$\frac{1}{4}$ VALVE

CHECK VALVE

6V

COVER

EMERGENCY POWER

VALVE

D.C POWER SUPPLY

AC POWER

CHECK VALVE

VALVE

MIXING TANK

LIQUID LEVEL CONTR (SINGLE ELEC.. EXP. PROOF

2-3 PSI N_2

VALVE

GAS DRIVEN MIXER

VALVE

CARBOY - (ANHYDRO ETHEL ETHER

RELIEF VALVE 3 PSI

(4) VALVES $\frac{1}{4}$

GLOVE BOX

TO VENT

NOTE:-
ALL VENT LINES TO CONNECT INTO COMMON LINE THROUGH ROOF HAVING FLAME ARRESTER AND PROTECTED FROM RAIN ETC.

CONDENSER

BUBBLER

RUPTURE DISC

VALVE

N_2 AR

2-3 PSI

EX. N_2-60-65 PSI $\frac{1}{4}$ VALVE

8-4

PROCESS FLOW DIAGRAM

60 PSI

MIXER - 60-65 PSI —

PURGES — 2-3 PSI —

FILTER — 6-10 PSI —

AGITATOR - 15-25 PSI —

NITROGEN BANK

65 PSI
(THREE) REGULATORS TO HAVE
BUILT-IN CHECK VALVES

(8) VALVES

PURGES - 2-3 PSI —

ARGON BANK

EMERGENCY DUMP

H TO DISCONNECT
T LEVEL CONTROL
FILTER IS NOT IN USE

CHECK
VALVE

LIQUID LEVEL   (SINGLE ELEC.
CONTROL   SWITCH)
                 EXP. PROOF

VALVE

QUICK
COUPLING

(5) VALVES

GE
NE

CONDENSER

HIGH LEVEL (ENERGIZES (2) WAY SOLENOID "A"
           AND DE-ENERGIZES (2) WAY SOLENOID "B"
LOW LEVEL (ENERGIZES (2) WAY SOLENOID "B"
          AND DE-ENERGIZES (2) WAY SOLENOID "A"

EXP. PROOF
(2) WAY SOLEN. "A"
NOR. CLOSE

— N₂

EXP. PROOF
(2) WAY SOLENOID "B"
NOR. CLOSED

— N₂
6-10 PSI

— TO VENT

RELIEF
VALVE ½ PSI

FILTER

LEVEL
CONTROL

SOLUTION
FROM TANK

VALVE

QUICK
COUPLING

WITCH)

CHECK
VALVE

SWITCH TO DISCONNECT
FLOAT LEVEL CONTROL WHEN
FILTER IS NOT IN USE

HIGH LEVEL (ENERGIZES (2) WAY SOLENOID "A"
           AND DE-ENERGIZES SOLENOID "B"
LOW LEVEL (ENERGIZES (2) WAY SOLENOID "B"
          AND DE-ENERGIZES SOLENOID "A"

N₂

HIGH LEVEL

6 IN.

LOW LEVEL

EMERGENCY DUMP

SOLUTION —
TO TANK

FIGURE 8-1

8-5

ASSEMBLY DRAWING OF PLATING TANK
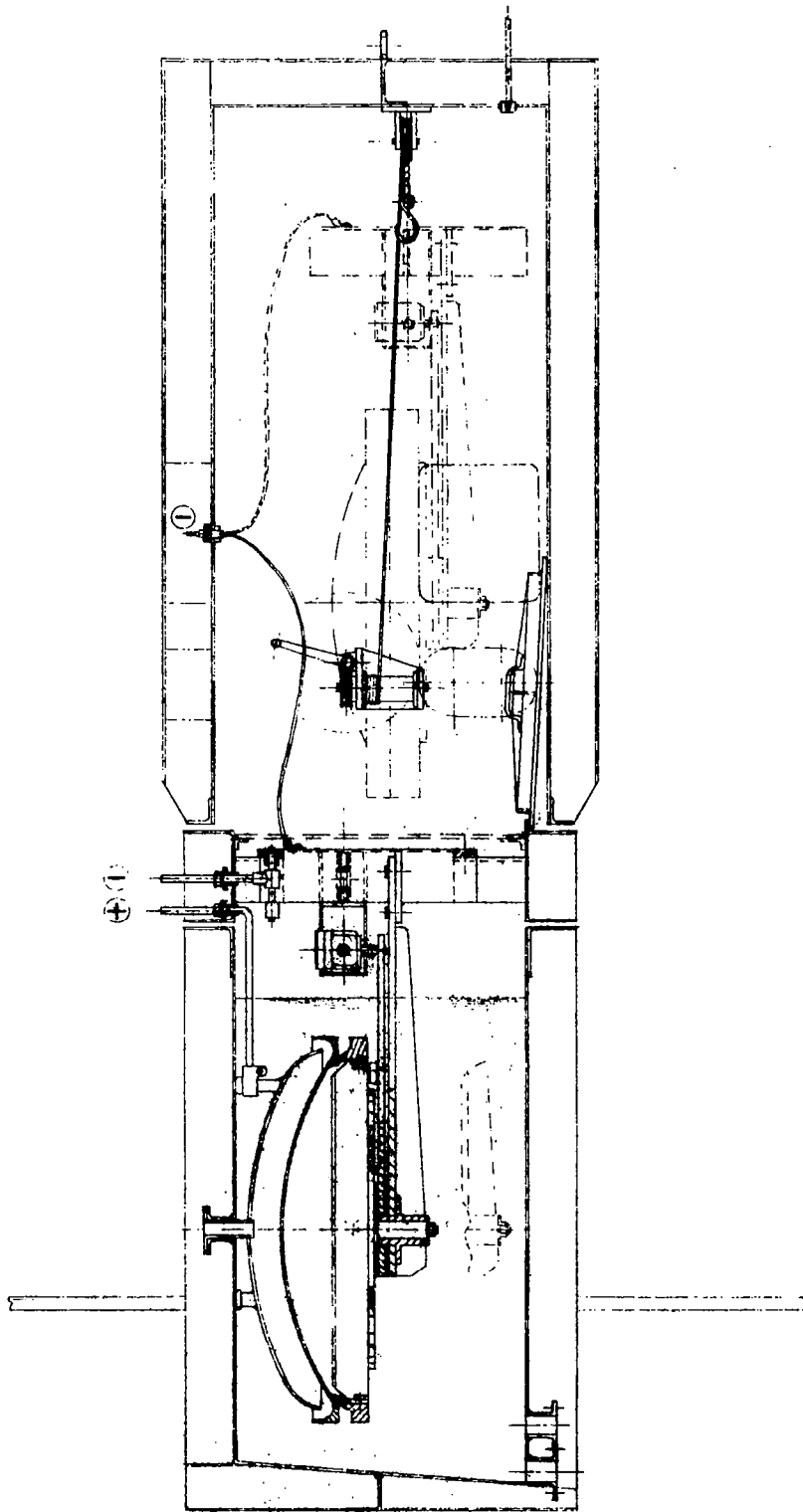
FIGURE 8-2
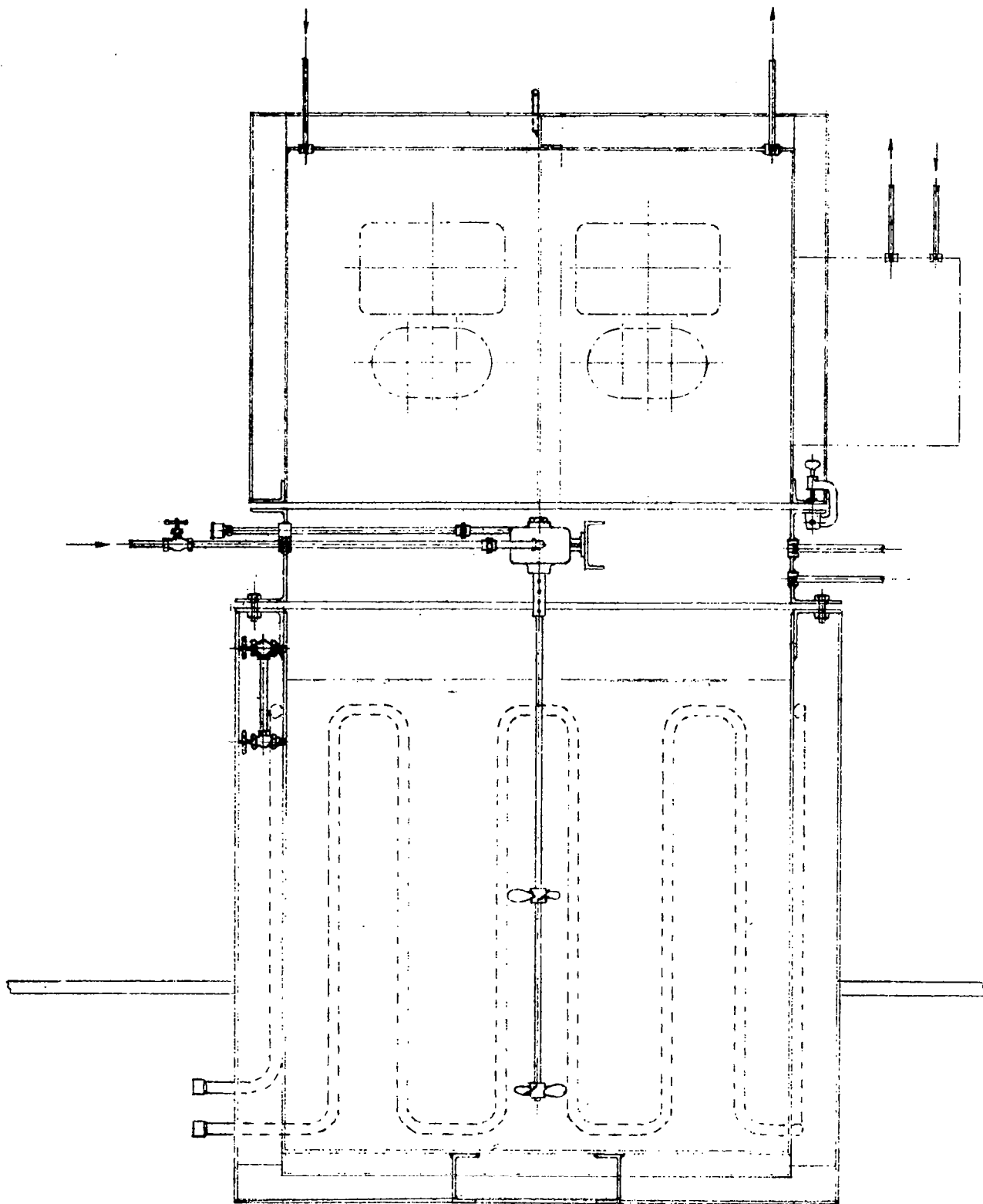
ASSEMBLY DRAWING OF STORAGE (MIXING) TANK
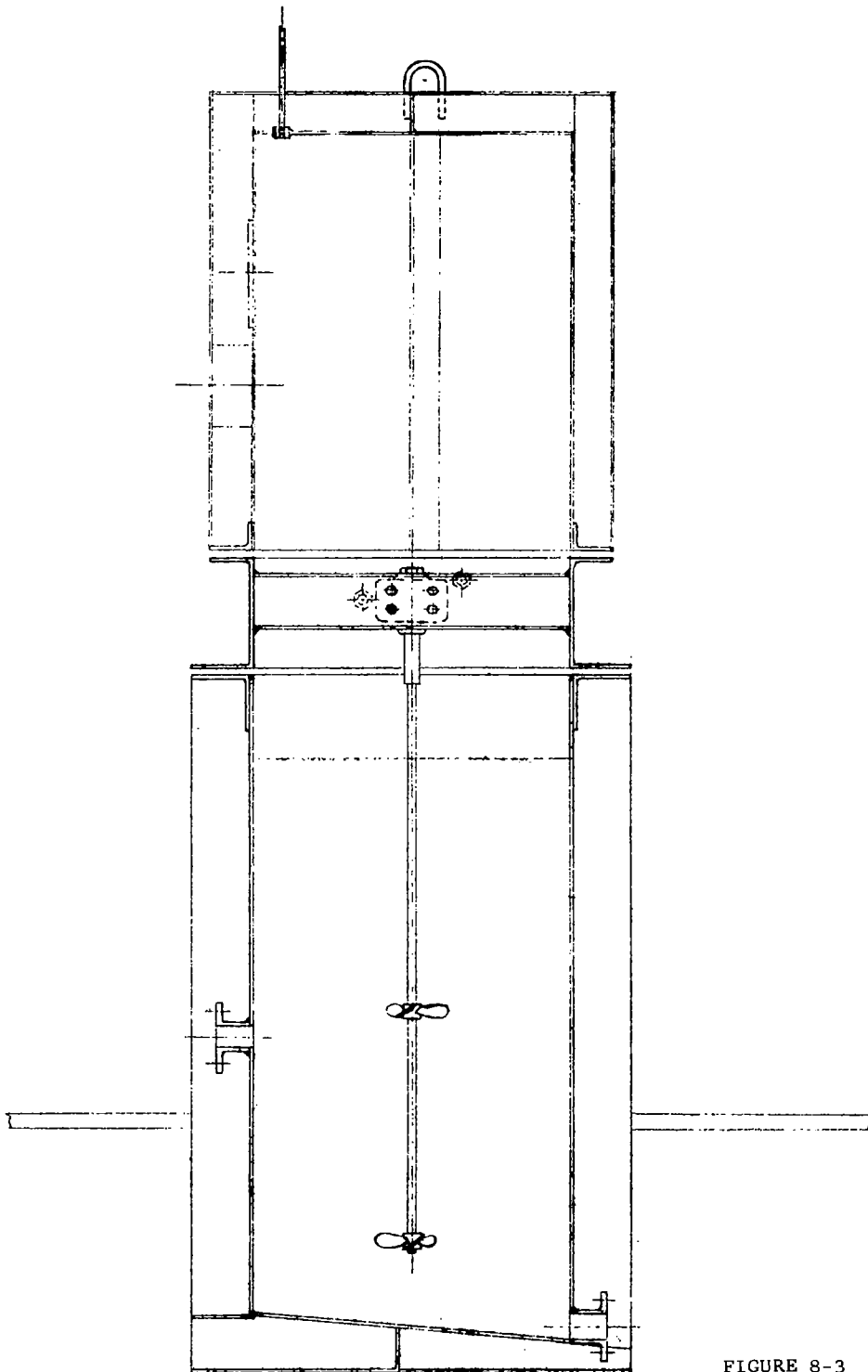
FIGURE 8-3

FILTER ASSEMBLY

1.22 Sq. Ft.
FILTER AREA

1 IN. VOL = 1.04 GALS.

SOLU.

FIGURE 8-4

8-11
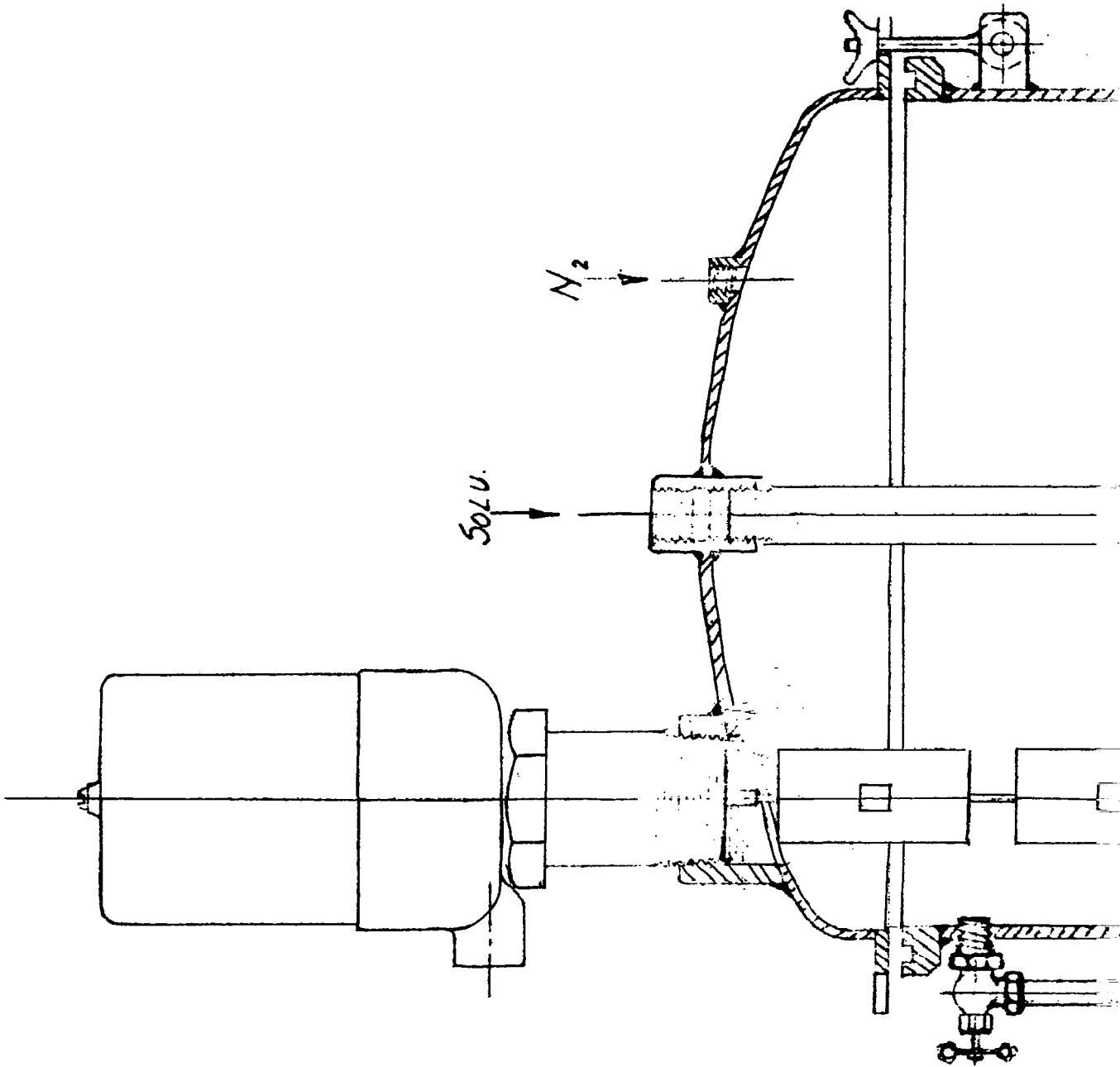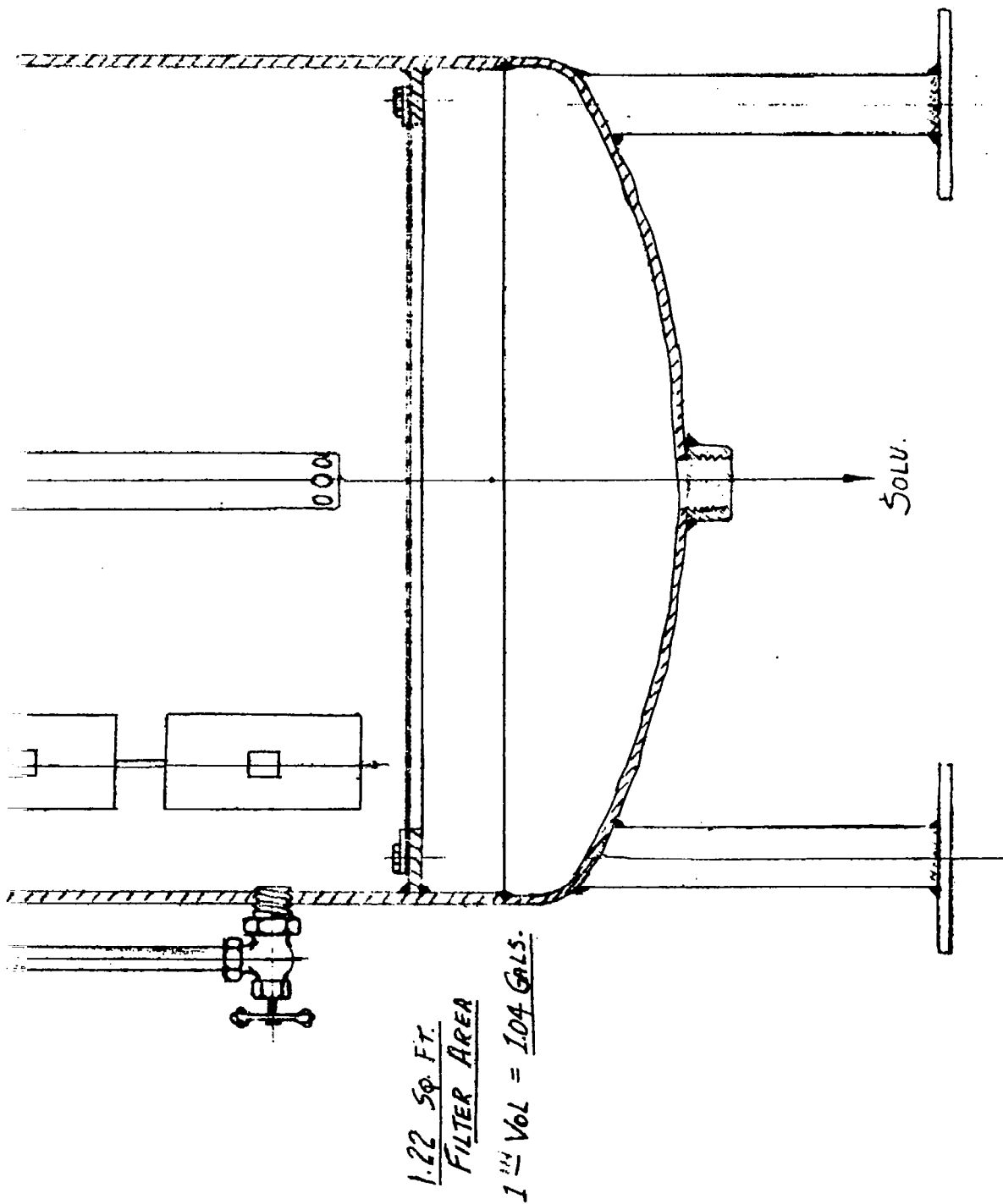
9. OPERATION OF THE ALUMINUM ELECTROFORMING EQUIPMENT

9.1   PREPARATION OF ALUMINUM PLATING SOLUTION

Anhydrous diethyl ether was transferred into the mixing tank, which was pre-
viously purged free of air and moisture, by pressurizing drums of ether with
dry nitrogen. The addition of aluminum chloride was made in small increments
while the solution was under constant agitation. Continuous cooling controlled
the solution temperature during the highly exothermic reaction of solution (580
calories per gram $AlCl_3$).

After the total solution of $AlCl_3$ (to a 3.4 molar concentration), the $LiAlH_4$
was added in small increments to obtain a 0.4 moles concentration. This latter
addition resulted in considerable gas evolution at first. Because of our labora-
tory experience and prior vendor information, this was anticipated and not re-
garded with any concern. The prepared solution was then filtered to remove un-
dissolved residues.

9.2   PLATING CELL

The conforming aluminum anode (see Fig. 9-5), properly shrouded with glass cloth
to retain the anode slime and minimize contamination of the plating solution, was
mounted in the empty plating tank. The solution return line from the filter passed
through a centrally located hole in the anode, such that filtered solution im-
pinged directly on aluminum deposit to cleanse the deposited surface. The plating
tank and its glove box enclosure were thoroughly purged with argon, and the
plating solution was pumped therein from the solution storage (mixing) tank through
the filtration system. The plating tank cover was clamped in position to prevent
exposure to the atmosphere when the glove box was subsequently removed. A slight
positive pressure (dry nitrogen) was maintained within the plating tank.

The glove box was then unclamped and removed, and the cathode assembly (see Fig. 9-2)
containing the male nickel master was attached to the hoist mechanism within the
glove box. The entire assembly (male master within the glove box) was then placed
in position over the plating tank, clamped down and purged thoroughly with argon.
Sparkproof electrical connections were made to the cathode and anode, and the
cathode assembly was quickly lowered into the plating solution subsequent to the
removal of the plating tank cover. Current density was then adjusted to 20 asf,
while plating solution circulated through the filter unit. Rotation of the master
began at this time. Figure 9-1 shows the relation of the entire assembly within
the plating tank.

The filter system functions identically to the laboratory filter and was described
in detail in a previous section. The scaled-up filter was completely automated,
using an explosion-proof flotation device regulating the upper and lower solution
levels within the filter.

A nitrogen-driven piston-displacement cylinder was used to move a ratchet which catches several gear teeth per displacement, resulting in a slow rotational movement of the male nickel master. Preventive maintenance on the cylinder between deposition runs averted any difficulties in actual operation.

9.3   OPERATING CONDITIONS DURING DEPOSITION (Design Goal)

The following plating parameters were used during the aluminum electroforming operation:

Anode-cathode	Parallel, at a distance of 2½", the anode remaining stationary.
Solution	Aluminum chloride (3.4M), lithium aluminum hydride (0.4M) in diethyl ether.
Temperature	Approx. 30°C (heating by the plating current)
Filtration	50 gal/hr (should be continuous)
Agitation	Agitation was provided by the filtering system, which impinged the solution on the cathode through a hole at the center of the anode. In addition, a slow rotational motion imparted to the male master provided additional agitation, and yielded a more uniform thickness in the 30" diameter aluminum electroform.
Periodic Reverse	A periodic current reversal cycle of: plating - 494 sec; deplating - 6 sec, was used. Additions during operation were made through the solution preparation tank.
Cell Voltage	15 - 20 Volts
Cell Current	150 to 200 Amps

9.4   TERMINATION OF THE DEPOSITION RUN

At the conclusion of the deposition period, the plating current, rotation and filtration were halted. The male nickel master with the adhering aluminum electroform (30" dia. mirror) was hoisted up into the glove box and adhering solution permitted to drain into the plating tank. The plating tank cover was replaced, and the glove box was thoroughly purged with argon to flush out all residual plating solution vapor. A positive pressure was maintained with the plating tank.

The glove box assembly was unclamped and lifted off the closed plating tank. The cathode assembly, containing the male nickel master with the aluminum electroform, was lowered and removed from the glove box, and subsequently set down near a water drain. The assembly was thoroughly rinsed with a heavy stream of water to react any adhering plating solution. The glove box assembly was returned to its position over the plating tank, clamped down, and a positive pressure re-established therein to prevent contamination of the plating solution by air or moisture.

9.5   ATTACHMENT OF MIRROR SUPPORT STRUCTURE

The mirror was electroformed with a peripheral grooved extension, anode guide ring, into which the torus ring could be placed (see Fig. 9-3). Subsequently, the ring and groove surfaces were thoroughly degreased and dried. The ring was centered inside the groove and bonded together with a low-shrinkage epoxy cement.

In electroforming one of the 30" diameter aluminum mirrors, the torus ring gutter was omitted, and the torus ring was bonded directly to the mirror. The bonding agent, in this instance, was a silicone rubber, specially formulated for this application.

A total of six runs were made, as detailed in the following section.

9.6   30" DIAMETER MIRROR DEPOSITION RUNS

9.6.1   INITIAL ("SHAKEDOWN") RUN

A "shakedown" run was scheduled so that all operational systems (filtration, rotation, purge, etc.) could be checked out simultaneously for this process. All systems functioned as designed without any operational difficulties. However, in the filters during the low pressure (or venting) cycle, ether from the plating solution was "flashed off" at a rate much greater than was anticipated or could be allowed, since continuous loss of ether would lower the plating solution level and expose a portion of the deposit. At best, the filtration unit could be operated intermittently. This observation was a serious blow, as lack of filtration would result in a roughened deposit for the 30" diameter aluminum electroforms, as compared with specimens prepared in the laboratory. Such roughness (nodules) will "show-through".

Corrective procedures, which could not be added at this late date, would be the design of a totally closed system; design of a filtration system different in principle, or installation of an ether recovery system to the filters, etc. As indicated, intermittent filtration was employed during the remaining deposition runs.

The aluminum electroform was too thin to separate successfully. Accordingly, the deposit was thermally shocked and lifted off. Separated in this manner, the mirror was literally turned inside out. It should be noted, however, that the electroform displayed a high degree of reflectivity and surface replication. The male nickel master was washed and prepared for the next run.

9.6.2   SECOND ELECTROFORMING RUN

The second deposition run was performed identically to the initial run, with an oleic acid-isopropanol solution sprayed on the surface prior to the final purge in the glove box. This run progressed without any difficulty and was terminated after the scheduled deposition time had elapsed.

The aluminum torus ring was bonded to the guide ring, as described in an earlier section. Moderate thermal shocking to separate the electroform from the male nickel master proved unsuccessful. Even dry ice was not sufficient to separate the mirror. This was identical to the effect of high oleic acid concentration in the laboratory runs, duplicated here, obviously, by the increased residence time, necessitated by the procedure for transfer of the master into the bath. Ultimately, the electroform was removed, although only by greatly distorting the mirror. The male nickel master was cleaned up and stored so that the adhesion, experienced on the male nickel master, could be investigated in the laboratory.

9.6.3   THIRD ELECTROFORMING RUN

After some laboratory experimentation, a proper balance between concentration and residence time was established. This necessitated application of the oleic acid-isopropanol solution immediately prior to the male master being lowered into the solution. This procedure was identically followed in all subsequent runs.

The run was terminated as scheduled, and the torus ring was bonded to the guide ring. Thermal shocking (dry ice and boiling water) was sufficient to separate the electroform locally, however, the mirror was still stuck at numerous points. In separating, minute holes were torn out of the electroform as the mirror was literally keyed into the microscopic numerous holes and pits. Critical examination of the male nickel master revealed the raised spots of aluminum embedded in these pores. Re-examination of the aluminum deposit from the first and second run indicated like holes. This phenomena was previously masked by the one "obvious" reason for failure to separate, too thin and overactivation by oleic acid, respectively. The cause, it was now realized, was twofold.

The deposition of aluminum into microscopically small holes, where the depth to diameter ratio in some measured locations was over 10, was deleterious for the present program, but at the same time, its significance, as regards electroforming in general, was realized. One of the greatest advantages of any electroforming, when compared to other fabrication processes, is the faithful reproduction of surfaces. The present ethereal aluminum process appears significantly superior to all other electroforming processes in this respect, thus we are dealing with a great improvement over an already excellent technique. The reason for such good penetration partially lies in the low viscosity and low surface tension of the ethereal solution, as compared to conventional aqueous processes.

9.6.4   FOURTH ELECTROFORMING RUN

An electroforming run, identical to the previous one, was performed without any problems during deposition.

The run was terminated on schedule, and an aluminum torus ring was again bonded to the guide ring, as described previously. To prevent distortion on separation due to the electroform interlocking in the many holes and pits, a solvent-soluble wax with a melting point of 220°F was applied to the electroform. It was hoped that the increased stiffness of the electroform (aluminum deposit plus adhering wax) would overcome the effect of keying in.

The aluminum deposit separated exactly as did the previous one - with numerous minute holes torn out of the electroform. The applied wax lifted away from the aluminum, such that no support or increased strength was obtained. Also, the thermal shock caused by the hot wax may have caused additional distortion. The male nickel master was cleaned again and prepared for the next electroforming run.

9.6.5   FIFTH ELECTROFORMING RUN

Since our difficulties were caused by the exceptional throwing power of the aluminum deposit penetrating the minute pits and holes, it was apparent that copper or other deposits would more readily separate. Accordingly, with NASA-Langley approval, a deposition run, in which a 0.001" copper flash would be deposited, upon which aluminum would be deposited (preceded by an oleic acid solution to promote adhesion between copper and aluminum) was scheduled. This proceeded as planned and, after removal from the plating bath, an aluminum torus ring was bonded to the guide ring. It should be noted that the mold surface was buffed before this run to remove marks of previous four separation tries. Polishing was held to a minimum. The electroform readily separated from the male nickel master, since the 0.001" copper flash did not key in, as had the previous all-aluminum deposits. The 30" diameter mirror was crated and stored for subsequent vacuum coating and inspection. The male nickel master was cleaned up in preparation for the next deposition run.

9.6.6   SIXTH ELECTROFORMING RUN

Additional laboratory investigation revealed another consequence of the excellent throwing power of the aluminum deposit. Aluminum, instead of bridging the junction of the guide ring knife edge and male nickel master (see Figures 9-3 and 9-4), deposits behind and underneath the knife edge. This results in a ridge or notch, which represents a weak or tear point. Also, in our laboratory runs, the edge of the aluminum deposit was broken away from the electroformed nickel tool to simplify separation of the electroform. The location of the guide ring (see Fig. 7-1c) was such that this was not possible. Consequently, for this new run, the guide ring was omitted and the aluminum torus ring was bonded directly to the electroformed mirror with a flexible RTV bond. Prior to deposition, the male master was mechanically polished again to reduce significantly the number of microscopic pits and minimize separation difficulties. Approximately $\frac{1}{2}$ mil surface was removed in this operation.

The male nickel master substrate was treated identically to the third and fourth electroforming runs (described earlier). An initial copper flash was not utilized in this run. Deposition proceeded routinely, and upon termination of the run, the male nickel master with the adhering aluminum electroform was removed from the bath, and the torus ring was attached to the mirror.

To overcome the distorting effects of the severely employed separation procedures (thermal shocking, or forcing off of the deposit by air pressure directed between master and electroform), a new separation procedure was tried.

The entire assembly, consisting of the male nickel master and the adhering aluminum electroform, were placed within a vacuum chamber to eliminate the force exerted by atmospheric pressure. Three spring devices imparting a combined lifting force of about 10 pounds were attached to the mounting holes in the torus ring. In this manner, the aluminum electroform was removed without difficulty.

The large size plating cell was placed on inactive status, and the plating solution maintained under a positive pressure of dry nitrogen.

9.7   ANODE GUIDE RING

9.7.1   DESIGN

It was the purpose of the anode guide ring to act as a mold for the forming of a transition piece between the active mirror skin and attachment point for the torus. It was designed such that any minor radial distortion of the torus or dimensional change at the bond line of the torus and transition piece would not be transmitted to the active portion of the mirror, namely, the 30" diameter portion.

The anode guide ring is fully described in Figure 9-3. This guide ring was used on the first five attempts of electroforming the aluminum skin, but was not used for the final mirror because of separation problems which occurred in the first four runs. All attempts to separate the mirror with the transition piece in place resulted in distortion of the mirror and transition piece. Furthermore, due to the excellent throwing power of the aluminum bath, the knife edge-to-master joint did not bridge well (the aluminum deposited underneath the knife edge, which resulted in a fault line).

9.8   TORUS

9.8.1   DESIGN

A single torus design, which would be bonded to a transition piece connecting the mirror to the bond area, was chosen. This torus is described in Figure 9-4. Because the mirror was not to be flight-weight, the torus was designed to be fabricated

from a standard aluminum pipe having an OD of 2.0 inches and a wall thickness of .028 inch. This was the lowest weight standard aluminum pipe available in this size, however, no vendor would undertake its fabrication with less than a 1/8" wall thickness. Therefore, a 1/8" wall pipe was rolled in the conventional manner, welded, and stress-relieved to form the torus. The excessive weight of the fabricated torus was reduced by a combination of drilling holes and chemically milling.

To form the three hard points required for mounting, three aluminum rods were inserted in the torus at three equal distances around the circumference. These rods were drilled and tapped for ¼" - 20 thread. These holes are located on the back side of the mirror and are parallel to its axis.

9.8.2   ATTACHMENT OF TORUS

As previously discussed in the section on the anode guide ring, it was intended to bond the torus to the transition section. When conditions made this undesirable, it was decided to bond the torus directly to the back surface of the mirror, inside the 30" diameter area.

Because this condition of bonding directly to the dish could cause possible distortions due to temperature gradients between the skin and the torus, the bonding agent used was a room temperature vulcanizing rubber. A thickness of approximately 1/8" was used. This type of bond will permit some relative movement between the skin and the torus without causing distortion to occur on the mirror. RTV has the added advantage of curing with essentially zero shrinkage.
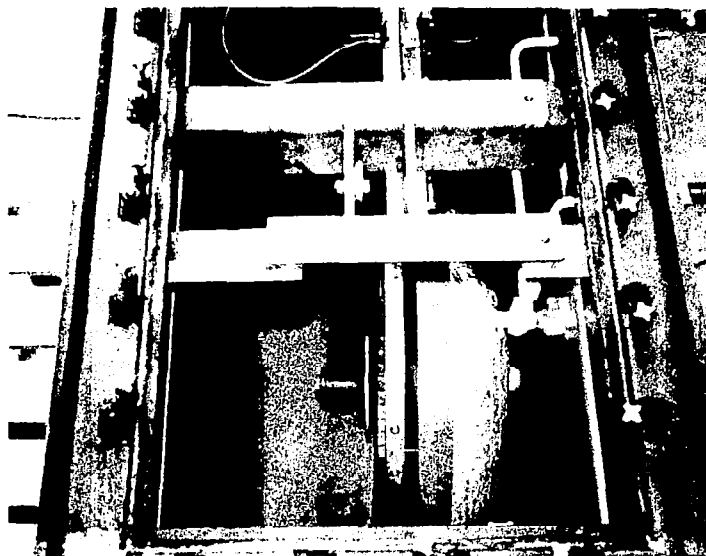
FIGURE 9-1 - CATHODE ASSEMBLY IN TANK

Looking down into plating tank. The
cathode assembly was lowered into the
plating tank in a dry run to check
clearances, etc. NOTE: glass cloth
covered anode on right. Progressing
to the left is the anode guide ring,
recessed portion of master, male mas-
ter support ring, ratchet gear, fixed
cathode assembly support. The recipro-
cating cylinder gas lines are visible
on top. Clamps hold down tank cover
within glove box.
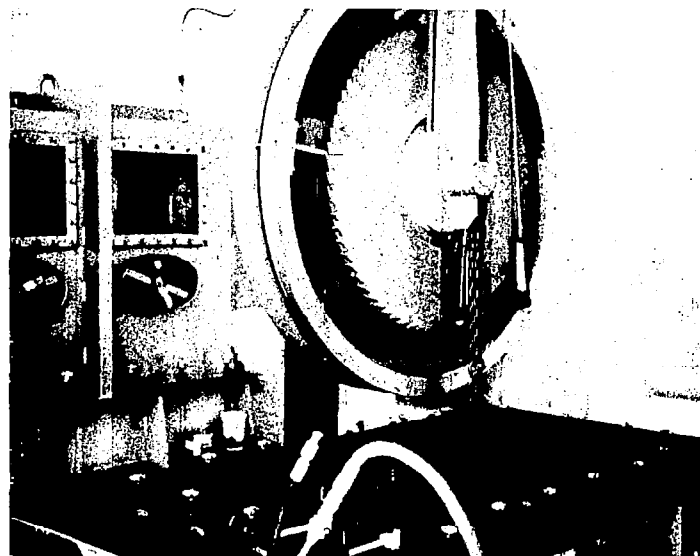
FIGURE 9-2 - CATHODE ASSEMBLY REVERSE
SIDE

Looking at the reverse side of the
cathode assembly. The three-legged
member (spider) is visible beneath
the gear. Glove box over storage-
mixing tank appears on left. The tip
of the ratchet is visible at the top
left of the fixed vertical member.

Section thru ℄

Mat'l
316 Staincess
Steel

See Full
Section

9-10

REVISIONS

SYM	ZONE	DESCRIPTION	DATE	APPROVED
A		(1) Added (6) 1/4-20 Tapped Holes	4/14/	

34 1/4 DIA.

31 DIA.

29 3/4 DIA.

13/16

2 3/8

.010

RAZOR
SHARP EDGE

20°

FULL SIZE SECTION

1/4-20 TAP - 7/8 DEEP - (6)
HOLES EQUALLY SPACED ON
33.375 DIA. B.C. WITHIN .010 T/P

SIZE
ZONE E.3

UNLESS OTHERWISE SPECIFIED	SIGNATURES	DATE	GENERAL ⊛ ELECTRIC
DIMENSIONS ARE IN INCHES	DRWN	5/13/6	DEPT LOC.
TOLERANCES ON Fractions ±1/32	CHECKED		TITLE ANODE GUIDE RING
2-PLACE DECIMALS ±	ISSUED		30" ALUMINUM MIRROR
3-PLACE DECIMALS ±	ENGRG		
ANGLES ±	MFG	5-13-6	
ALL SURFACES ✓	MTLS		CONTRACT NO.
MATERIAL -			CODE IDENT NO. SIZE DWG NO.
GOVT OR COML			D SK5-0258
QE	FIGURE 9-3		SCALE 1/2 NC NOTED WT CALC ACTUAL SHEET 1 OF 1

SK5-0258

G

F

E

D
FOLD

$32\frac{5}{8}$ REF

C

201

1   Mirror (As Formed)

B

SECTION THRU ℄

A

SYM	ZONE	REVISIONS		
		DESCRIPTION	DATE	APPROVED

G

F

E

D

$5R5-0252$

SIZE D

REV

FOLD

B

$\frac{1}{8}$ NOM (GAP FOR BONDING AGENT)

.030 (REF.)

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES— TOLERANCES ON FRACTIONS ± 1/32 2-PLACE DECIMALS ± 3-PLACE DECIMALS ± ANGLES ± ALL SURFACES √ MATERIAL— GOVT OR COML— GE—		SIGNATURES	DATE
	DRAWN		5/4/66
	CHECKED		
	ISSUED		
	ENGRG		
	MFG		
	QUAL		

GENERAL ⊛ ELECTRIC

SPACECRAFT — DEPT LOC VALLEY FORGE

TITLE
TORUS SUPPORT
FOR
30" DIA. ALUMINUM MIRROR

CONTRACT NO.

CODE IDENT NO	SIZE	DWG NO.
	D	$5R5-0252$

SCALE 1/2 SIZE | WT CALC ACTUAL | SHEET 1 OF 2

FIGURE 9-4

G

F

E

30°
REF.

D
FOLD

C

WELD
GRIND FLUSH
TYP (2)
PLACES

B

A∩T₁

$32 \frac{5}{8} \pm \frac{1}{16}$ DIA.

A

4   3 FOLD   2   1

REVISIONS

SYM	ZONE	DESCRIPTION	DATE	APPROVED

¼-20 TAP ½ DEEP
(3) HOLES EQUALLY
SPACED WITHIN .010
ON A 30⅝ DIA. B.C.

D SK5-0252

FILLET WELD
ALL AROUND
TOP & BOTTOM

202	3	6061-T6 ALUM.	½ DIA. x 2⅜ LG.	INSERT
201	1	ALUM (6061-T6) TUBING	2 O.D. x .028 WALL x 14⅝ LG	SUPPORT RING
ITEM	QTY	MAT'L	ROUGH SIZE	DESCRIPTION

BILL OF MATERIAL

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES— TOLERANCES ON: FRACTIONS ±1/16 2-PLACE DECIMALS ± 3-PLACE DECIMALS ± ANGLES ± ALL SURFACES ✓	SIGNATURES	DATE
MATERIAL—	DRAWN	5/4/64
GOVT OR COML	CHECKED	
	ISSUED	
GE	CHECKED R. W. Meyers	8/6/64
	MFG	

GENERAL ⊕ ELECTRIC
SPACECRAFT DEPT LOC. VALLEY FORGE

TITLE
DETAIL
TORUS SUPPORT RING 30 DIA MODEL

CONTRACT NO.

CODE IDENT NO.	SIZE	DWG NO.
	D	SK5-0252

| SCALE | WT CALC ACTUAL | SHEET 2 OF 2 |

FIGURE 9-4

30^R

INSIDE CORNER

$30\frac{3}{4}$

$5\frac{8}{}$

6
INSIDE

$\frac{5}{8}$

$30°$

5^R

$1\frac{1}{2}$

(3) STUDS (DETAIL "A")

(ALUMINUM WELD AFTER MACHINING)

$\frac{1}{4}$ MATERIAL THICKNESS

$30°$

$\frac{1}{32}R$

$\frac{3}{4}$

2

$3\frac{7}{8}$ FIT AT ASSEM.

DETAIL "A"
FULL SIZE

CHANGE LETTER	DESCRIPTION	DRFT.	DATE

Drill Amt.	Anodize Data Face To Face
32	3" FROM
32	2½ "
28	3 "
24	3⅜ "
20	3⅛ "
14	2⅝ "
8	1¹⁴⁄₁₆ "

30°

30°

2³⁄₁₆ DIA.

25 DIA.

FIGURE 9-5

CONFORMING ALUMINUM ANODE

9-17

10. 30-INCH ELECTROFORMED ALUMINUM MIRRORS

Two of the six deposited 30" diameter electroformed aluminum mirrors served to
fulfill the contractual requirements. One aluminum mirror has a thin (approxi-
mately 0.001") copper flash on the reflecting surface, deposited to facilitate
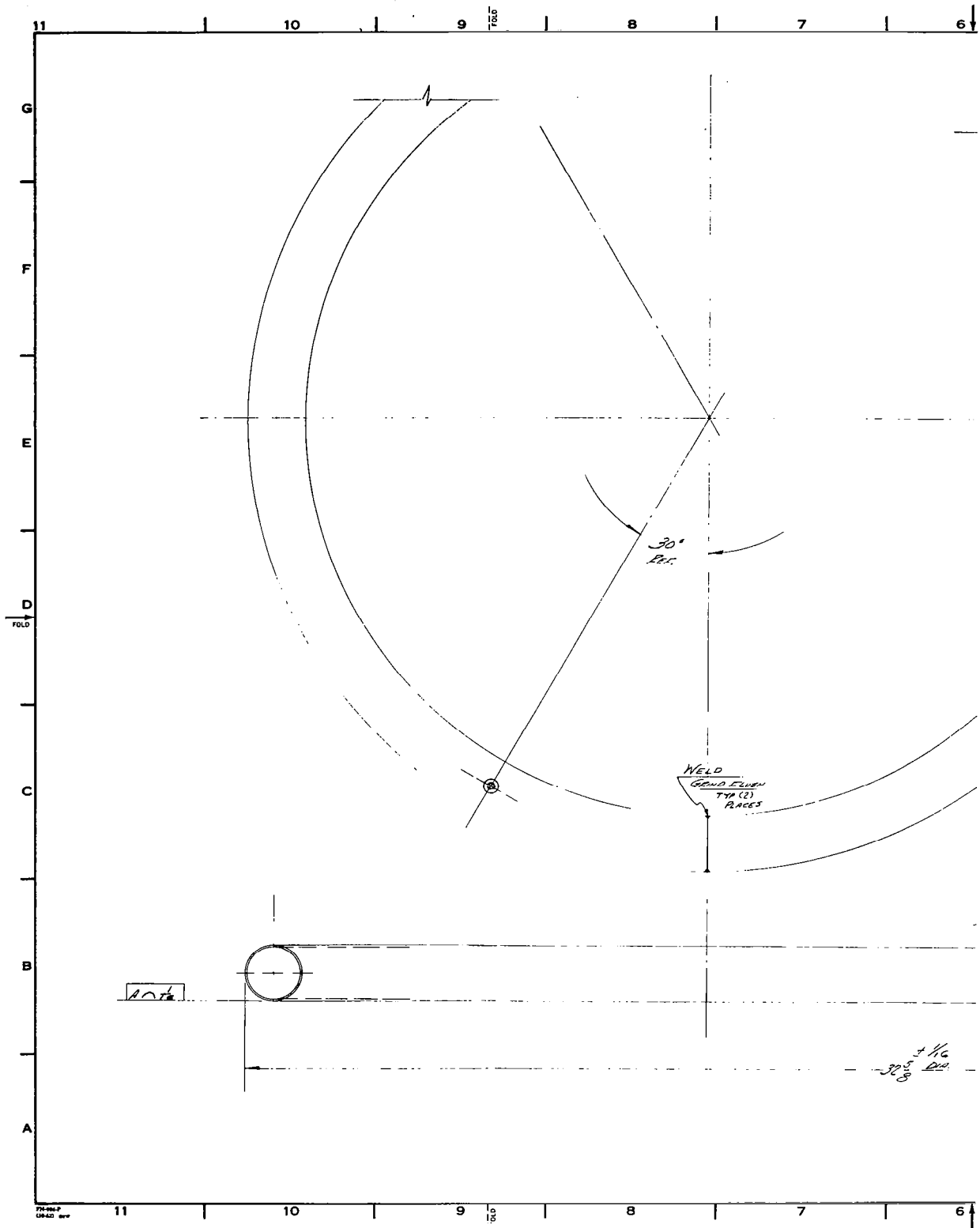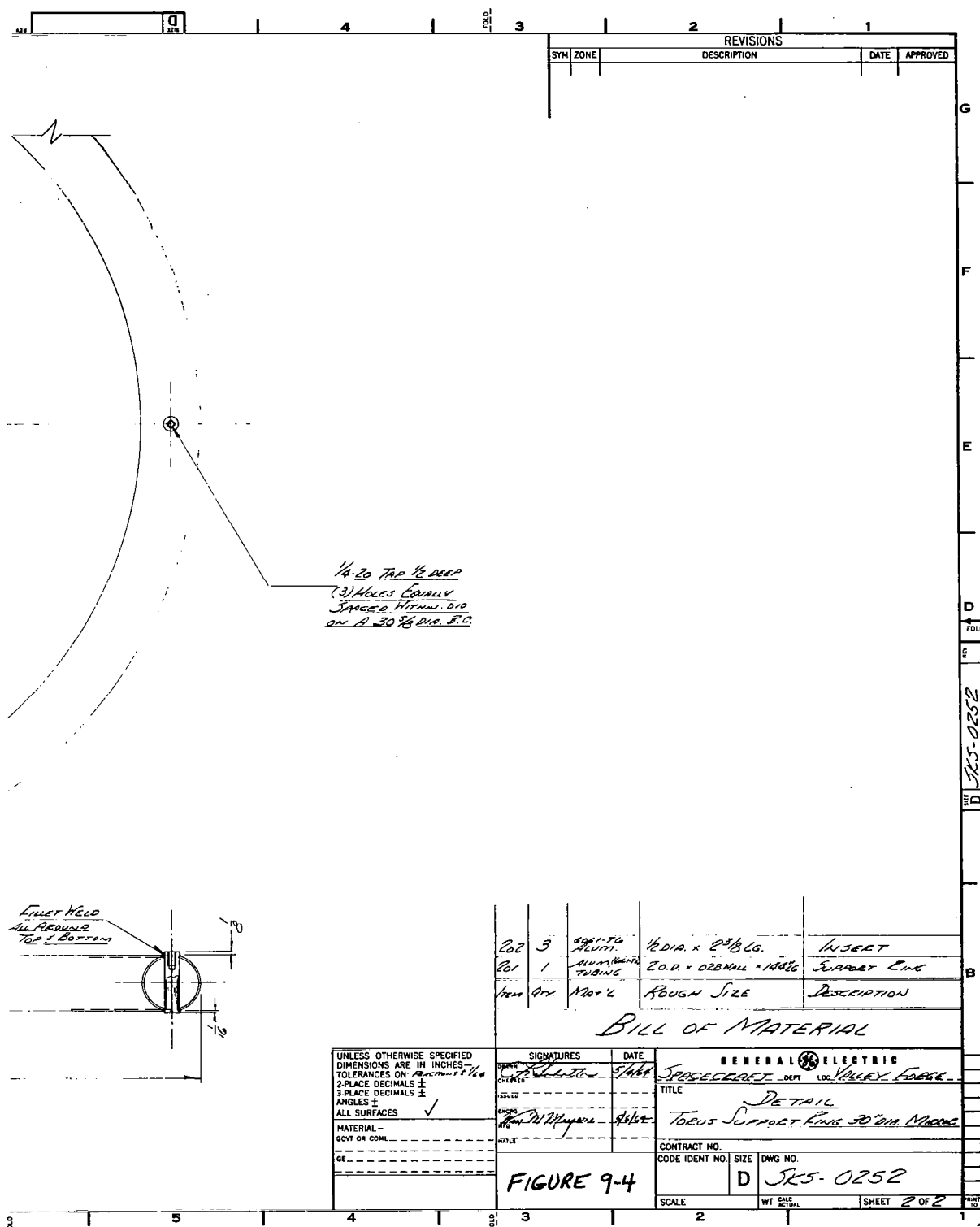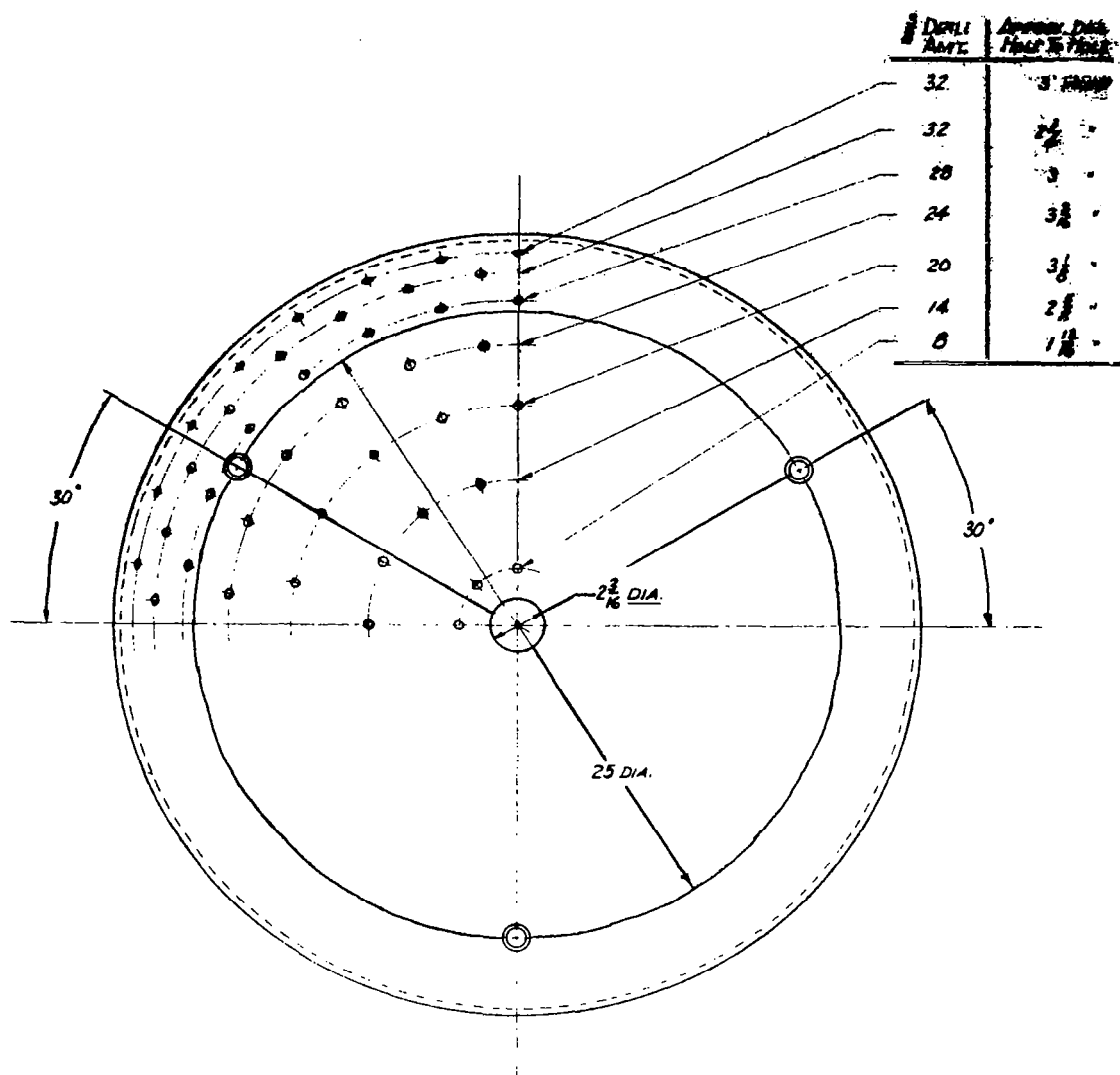separation from the nickel master. The bulk thickness of electroformed alumi-
num is electrochemically bonded to the copper skin. The other mirror was all-
aluminum. This mirror is shown in Figure 10-2. Their fabrication was described
in sections 9.6.5 and 9.6.6, respectively. Weight of the mirrors appear in
Table 10-1.

10.1   VACUUM COATING

The two electroformed aluminum mirrors were transported to a subcontractor for
vacuum coating. In the vendor's vacuum chamber, a thin aluminum and silicon
monoxide film was deposited successively on the reflective surface of the mirrors.
The quality of the vacuum coating was far below that for the optical test speci-
mens described in section 5. The coating "bloomed" in a segment of the mirrors,
imparted a haze or mist-like character to the reflective surface and was flaky
(can be blown off) in several regions. Questioned about the quality of the
vacuum coating, the subcontractor's representatives admit this phenomena has
occurred lately even with bare nickel substrates. They have no explanation at
this time, and have indicated that they plan to investigate the situation and
will recoat these two concentrators free of charge, if required. Their report
is forthcoming. It should be noted that the vacuum coating of the aluminum
test specimens, which are still highly reflective after 6 months, are without
any hint of degradation. (Since the subcontractor regards the process proprietary,
GE/RSD could not investigate the problem directly.) The two mirrors were then
transported back to Philadelphia for optical inspection of the geometric accuracy
of the surface.

10.2   OPTICAL INSPECTION

Each mirror was mounted on a rotary table to facilitate multipoint inspection.
An optical bar was positioned horizontally over the mirror. Two plunging-type
theodolites were mounted on the optical bar, and the mirror was inspected verti-
cally, after initially locating and placing a "point source of light" at the
focal point. The procedure was described in greater detail in section 6. The
tabulated inspection data appear in Tables 10-2a and 10-2b.

The two electroformed aluminum mirrors had average slope errors of 6.9 minutes
and 9.4 minutes for the copper-flashed and all-aluminum mirror, respectively.
The increased slope error, as compared with that for the spincast epoxy female
mold (0.66 minutes), resulted from local distortion caused by interlocking of
the electroform into microscopic holes in the male nickel master, show-through
of nodules resulting from insufficient agitation during deposition, and from

the mechanical polishing of the male nickel master which was necessary to
minimize "keying-in" of the deposit and to permit distortion-free separation
of the electroform. Despite the mechanical polishing of the male master, some
mechanical interlocking of the all-aluminum electroform occurred, which is re-
flected in a greater slope error than that for the copper-flashed mirror.

10.3   MIRROR ANALYSIS

An analysis for the problem of stress and deformation of a paraboloidal shell
of revolution subject to a uniform gravity field parallel to its axis was per-
formed. This simulated a thin shell, supported by a torus, under launch loads.
Because a pure membrane solution cannot be obtained for this shell, the present
solution was achieved in two steps, i.e. membrane plus bending solutions.

The detailed analysis (reported in Appendix C) was programmed for an IBM 7094
digital computer. Slope rotations, meridional and circumferential stresses
were calculated for aluminum paraboloidal collectors of 30" diameter and 60°
rim angle having thicknesses of 0.010, 0.020, 0.030 and 0.040 inch. The col-
lectors were assumed fully fixed at their outer peripheries, i.e. no displace-
ments or slope rotations were permitted. This corresponds to the practical
case in which a support torus is connected to the outer periphery of a collec-
tor with the torus being much stiffer than the collector shell.

The results are presented in Tables 10-3 and 10-4 and in Figure 10-1. As is
apparent from Table 10-3 and Figure 10-1, the slope rotations are extremely
small, and they occur over the area adjacent to the support torus only. The
thinner the collector membrane, the smaller the affected area, but the larger
the maximum sloper rotation. A thickness of .020 or .030 inch appears best
from Figure 10-1, with the maximum slope rotation not exceeding .12 minutes,
i.e. 7 seconds of arc. If the collector is made thicker, the maximum slope
rotation is slightly reduced, but the area of slope deviation is considerably
enlarged. If the collector is made thinner, the maximum slope rotation rises
rapidly, and the area of slope rotation is decreased. Either of these changes
probably results in a decreased geometric collector efficiency.

Table 10-4 shows that the stresses under a 10g axial load condition are very
small. The maximum stresses are practically independent of the collector
thickness; differences showed up in the third significant figure only. The
maximum meridional stress occurs at the outer rim, and is equal to 32 psi. The
maximum circumferential stress equals 14 psi and occurs close to the outer
rim; it moves inward with increasing collector thickness.

The independence of the maximum stresses from the collector thickness can be
explained as follows. The membrane stress is obviously independent of the
thickness because weight, load and resisting area all vary linearly with the

thickness (see Fig. 10-1), but smaller moments only are required to cause these increased deformations as the thickness decreases. Similarly, the section modulus becomes smaller as the thickness decreases. The sum of these effects is an insignificantly small change in maximum bending stress over the thickness range investigated.

FIGURE 10-1

SLOPE ROTATION OF PARABOLOIDAL COLLECTOR
UNDER 10g AXIAL LOAD
FULL EXTERIOR RESTRAINT

Diameter: 30 in.
Rim Angle: 60 degrees
Slope Angle θ: 30 degrees
Collector Material: Aluminum
Collector Thickness: .010, .020, .030, .040 inch.
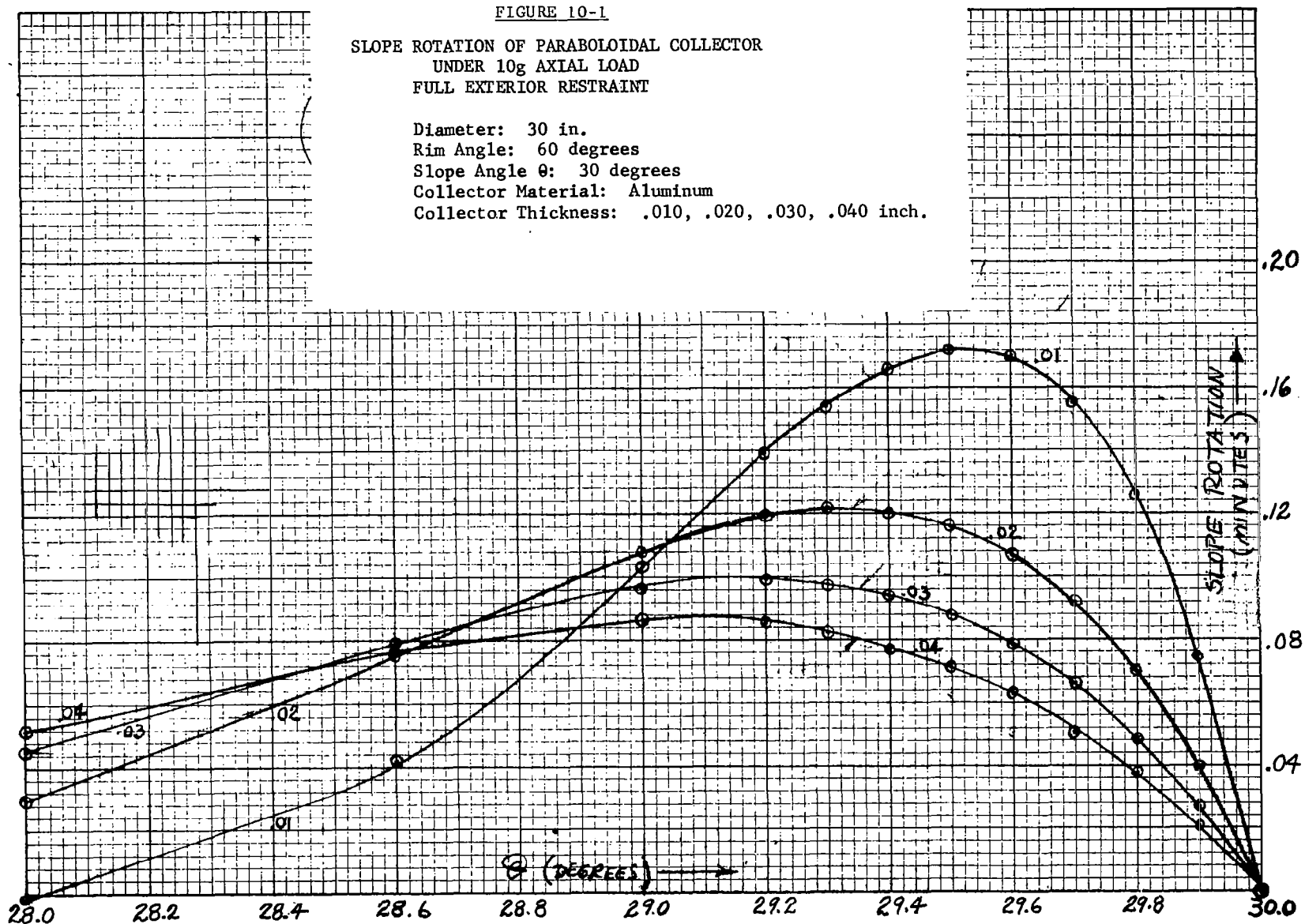
FIGURE 10-2

RUN 6 MIRROR

TABLE 10-1

WEIGHT OF ELECTROFORMED ALUMINUM MIRRORS

	Total Weight (lbs)	Weight per Unit Mirror Surface (lbs/ft^2)	(Kg/M^2)
RUN 5 MIRROR			
Total Assembly (mirror, gutter, torus, and bonding material)	3.45	0.705	3.46
Mirror and Gutter, alone	1.95	0.348	1.72
RUN 6 MIRROR			
Total Assembly* (mirror, torus, and bonding material)	3.44	0.616	3.02
Mirror, alone	1.44	0.256	1.25

* This mirror was electroformed without a gutter. The torus ring is bonded directly to the mirror.

TABLE 10-2A

ADJUSTED SLOPE ERRORS (MINUTES)

RUN 5 - 30" aluminum mirror (with copper flash)   Focal point: 13 ± 1/16 inch

Angular Station*	INSPECTION DIAMETER (INCH)				
	10	16	22	26	Average
A-1	1.8	8.6	2.2	17.7	7.7
A-2	2.3	1.2	2.3	11.4	4.3
A-3	1.8	1.9	5.8	8.3	4.5
A-4	3.1	2.4	7.8	17.9	7.8
B-1	3.7	5.4	9.3	6.5	6.2
B-2	6.4	5.4	3.8	7.5	5.8
B-3	6.2	7.1	15.1	12.2	10.1
B-4	1.4	10.0	14.9	21.0	11.8
C-1	1.9	4.2	13.3	23.2	10.7
C-2	2.7	4.3	4.4	9.3	5.2
C-3	2.4	4.2	7.6	4.0	4.0
C-4	5.8	10.8	0.6	5.4	4.7
AVG	3.3	4.7	7.3	12.3	6.9

* Refer to Figure 6-4

TABLE 10-2B

ADJUSTED SLOPE ERRORS (MINUTES)

RUN 6 - 30" aluminum mirror   Focal point: 13 ± 1/16 inch

Angular Station*	INSPECTION DIAMETER (INCH)				Average
	10	.16	22	26	
A-1	11.5	13.8	10.6	12.2	12.0
A-2	14.8	12.6	10.1	5.1	10.6
A-3	9.4	12.9	11.0	8.7	10.5
A-4	9.0	9.5	7.9	12.9	9.8
B-1	5.1	5.2	11.9	12.7	8.7
B-2	2.3	3.2	11.4	15.3	8.1
B-3	1.2	6.3	11.9	16.1	8.9
B-4	3.9	4.6	5.5	9.7	5.9
C-1	3.5	2.2	8.0	13.5	6.8
C-2	2.1	5.6	9.3	9.3	6.6
C-3	5.9	11.3	14.0	14.2	11.3
C-4	10.1	6.1	14.4	21.7	13.1
AVG	6.6	7.8	10.5	12.6	9.4

* Refer to Figure 6-4

TABLE 10-3

SLOPE ROTATION OF PARABOLOIDAL COLLECTOR
UNDER 10g AXIAL LOAD
FULL EXTERIOR RESTRAINT

Diameter: 30 in
Rim Angle: 60 degrees

Angle θ (degrees)	SLOPE ROTATION (MINUTES OF ARC)			
	COLLECTOR THICKNESS (IN.)			
	.010	.020	.030	.040
30.0	.000	.000	.000	.000
29.9	.075	.040	.027	.021
29.8	.125	.070	.049	.038
29.7	.155	.092	.066	.051
29.6	.170	.107	.079	.063
29.5	.172	.116	.088	.071
29.4	.166	.121	.094	.077
29.3	.154	.122	.098	.082
29.2	.139	.119	.099	.085
29.0	.103	.108	.097	.086
28.6	.042	.075	.079	.077
28.0	-.001	.029	.045	.052
27.0	-.005	-.003	.007	.016
25.0	-.000	-.002	-.004	-.004
20.0	-.001	-.001	-.001	-.001
10.0	-.001	-.001	-.001	-.001

R_o = 26.0 in.
θ_1 = 30 degrees
= 10
Material = aluminum

TABLE 10-4

MAXIMUM STRESSES IN PARABOLOIDAL COLLECTOR
UNDER 10g AXIAL LOAD
FULL EXTERIOR RESTRAINT

Diameter: 30 inch.
Rim Angle: 60 degrees

Collector Thickness (in.)	.010	.020	.030	.040
Maximum Meridional Stress (psi)	32	32	32	32
Location (θ, degrees)	30	30	30	30
Maximum Circumferential Stress (psi)	14	14	14	14
Location (θ, degrees)	28	27	25	25

NOTE: The stresses indicated are the maximum total stresses (membrane plus bending)
at the concave surface of an aluminum collector.

REFERENCES

The following is a list of references containing information pertinent to the Aluminum Electrodeposition Program.

(1)   Deville, H. St-C., Chemist, 13, No. 2, 12 (1854).

(2)   Plotnikov, V., J. Russ. Phys. Chem. Soc., 34, 466 (1902).

(3)   Safranek, W.H., Schickner, W.C. and Faust, C.L.; "Electroforming Al Waveguides Using Organo-Al Plating Baths"; J.E.S., 99, p 53-9 (1952).

(4)   Couch, D.E. and Brenner, A.; "A Hydride Bath for the Electrodeposition of Aluminum"; J. Electrochem. Soc, 99, p 234-244 (1952).

(5)   MacMullin, R.B.; "The Problem of Scale-Up in Electrolytic Processes", Electrochem Technology, 1, No. 1-2, Jan-Feb 1963.

(6)   Alm, G.V. and Binstock. M.H.; "Aluminum-Electrocladding Studies for Proposed OMR Fuel Elements"; NAA-SR-2704.

(7)   Evans, G.G., Kennedy, J.K. and DelGreco, F.P.; "Conductometric Study of the Reactions Between Metal Halides and Lithium Aluminum Hydride"; J. Inorg. Nucl. Chem., 4, p 40-47 (1957).

(8)   Connor, J.H. and Brenner, A.; "Electrodeposition of Metals from Organic Solutions"; J. Electrochem. Soc., 103, p 657-662 (1956).

(9)   Beach, J.G. and Faust, C.L.; "Electroclad Aluminum on Uranium"; J. Electrochem. Soc., 106, p 654-659 (1959).

(10)   Hurley, F.J. and Wier, T.P. Jr., "Electrodeposition of Metals from Fused Quaternary Ammonium Salts", J.E.S., 98, p 203-6 (1951).

(11)   Ziegler, L. and Gellert, H.G.; "Process for the Production of Pure Al"; US Patent 2,843,474 (1958).

(12)   Bunsen, R., Poggendorf's Ann., 97, p 648 (1854).

(13)   Ziegler, K. and Lehmkuhl, H.; "Process for the Electrolytic Deposition of Al"; US Patent 2,849,349 (1958).

(14)   MSD Patent Docket No. 35-28D-1410, F.J. Schmidt (1963).

(15)   "9½ Foot Diameter Master and Mirror", GE-MSD 64SD540, 20 March 1964.

WORK STATEMENT FOR ELECTROFORMING OF NICKEL MALE MASTER

The vendor will electroform a nominally 3/8 inch thick, 30 inch diameter, nickel master over the spincast epoxy mold, using the best up-to-date practices. The following steps will be used, except when variations are approved in advance by the RSD Project Engineer. RSD's representative will be present during the entire sequence and jointly with the vendor's representative will agree on and direct all process details. Final responsibility will rest with RSD's Project Engineer.

a. The spincast epoxy assembly will be prepared for the electroforming sequence by protecting all areas which should not be plated with masking compounds.

b. The working surface of the epoxy mold will be cleaned by spraying with mild alkaline cleaning and detergent solutions at room temperature. This cleaning step will be continued until a breakfree water film is observed over the entire working surface when distilled water rinse is sprinkled over the working surface.

c. The surface will be sensitized using a slightly acidic filtered stannous chloride sensitizing solution not over three hours old.

d. The sensitizing step will be followed by a sprinkled distilled water rinse.

e. The working surface will now be silverized by chemical reduction of silver from an ammoniacal silverizing solution. The ammoniacal silver and the reducer solutions (temperature not exceed 70°F) will be sprayed simultaneously over the working surface, and this spraying will continue until the silver deposit will become uniformly bright over the entire working surface.

The distilled water spray will be maintained over the surface during steps (a) through (e), to maintain the breakfree water film condition once established, but the amount of such spray will be decreased during the sensitizing and silverizing steps. The temperature of neither the master nor any of the solutions shall exceed 75°F during the steps (a) through (e).

f. The assembly will be transferred without delay into a 100°F nickel electroforming bath, and the electrolysis will start as soon as it is possible to establish correct positioning of the electrodes and contacts. The initially low CD will be gradually raised to full working value as specified below.

g. The nickel anode assembly will be constructed in such a manner as to provide a highly uniform current density distribution over the cathode.

h. The electrolyte will consist of a low stress Ni-sulfamate solution.

As soon after immersion of the mold assembly as equilibrium conditions are established, the following indicative parameters will be maintained.

Temperature: $120^{\circ}F \pm 1^{\circ}$
Current Density: 20 asf
Agitation: Impinging solution
Filtration: Continuous
Electrolytic Purification: Continuous
Controls: 1.   Essentially continuous monitoring of bath composition and pH.
2.   Continuous monitoring of temperature, current and voltage.
3.   Daily check of stresses using a Schmidt-cell.
4.   Surface tension.

i.   The spincast mold to be furnished by General Electric is fully described on GE Dwg. 243R306. All metal parts on this structure are fabricated from 300 series stainless steel, and will, therefore, not contaminate the bath.

j.   Also furnished by General Electric will be a stiffening ring structure which is to be attached to the electroform by electroforming during the process. This ring is described on GE Dwg. SKS 0250. All necessary cleaning and clamping of this ring to the spincast is to be performed by the vendor. GE will monitor the attaching of the GE-furnished stiffeners which are mounted to this ring prior to removing the electroform from the spincast. These stiffeners are also described on GE Dwg. SKS 0250.

A guide ring, described on GE Dwg. SKS 0250, will be furnished by GE and is to be assembled to the male master support structure under the supervision of GE engineering. This ring contains its own clamps so that no external clamping is required.

k.   At the end of the nickel electroforming process, when the proper deposit thickness is obtained, the assembly will be removed from the plating tank, and the bath residues will be rinsed off with de-ionized water.

Separation of the electroform will be effected by alternate expansion and contraction of the electroform, obtained by alternate hot and cold rinses and will be assisted by forcing 5-10 psi compressed air between the electroform and mold and by mechanical forces applied to the attachments.

WORK STATEMENT FOR
THE FABRICATION AND INSTALLATION OF
ALUMINUM ELECTROFORMING EQUIPMENT

The vendor will design, fabricate and install aluminum electroforming pilot equipment and appurtenances, as described below. Quote each of the listed tasks separately. The use of second-hand or rented equipment is acceptable, if such equipment fully satisfies the requirements contained herein.

1. Design, and after approval of the design by the Project Engineer, fabricate one aluminum electroforming pilot plant equipment and appurtenances in strict conformance with attached drawing and specifications. Any departure from the indicated details will require prior approval of Re-Entry Systems Department's Program Manager or Project Engineer. Design consultation will be furnished by RSD throughout the entire design and fabrication period.

2. After approval of the fabricated or procured components by RSD's Project Engineer, transport, assemble and install the equipment at a location provided by the vendor; equipment must be capable of electroforming up to 1/8" thick, 30" diameter paraboloidal aluminum mirrors over nickel master, which will not be more than 33" in diameter or more than 12" in depth, including substructure; and the equipment must remain in good operating condition for the duration of at least two (2) such electroforming runs. In addition to conforming to safety requirements imposed by handling of ether and hydride mixture, the electroforming area must provide cold running water, water drains and sufficient electrical outlets to satisfy the current requirements indicated in the specification.

3. Checkout the installed equipment to demonstrate capability for conformance with described process Flow Sheet and specifications. Both tank and the piping, filters, glove boxes, shall be sufficiently gas tight so that a 2.5 psi N_2 pressure over a period of 30 minutes shall not drop below 2.0 psi. All cooling piping, valves and filters must not show any sign of leakage.

The time for the design, fabrication, installation and checkout of the facility shall not exceed 3 1/2 months, starting from the date of receipt of authorization for go-ahead.

4. Two carbon steel tanks, of suitable size to accommodate the immersion of the 33-inch diameter x 12-inch deep nickel master, must be provided in an area adjacent to the electroforming equipment. These tanks will be utilized during the surface preparation cycle, immediately prior to electroforming. Electrical connections to these tanks are not required; however, tank drainage must be provided. All solutions will be supplied by RSD.

5. The electroformed nickel master, anodes, anode bags, electrode connection from tank to within glove box, all chemicals necessary for the preparation of the plating bath, and a periodic reverse timer unit will be supplied by RSD. All other items, indicated in the specifications and drawings, including a current regulated (±1%) DC and an AC power source, connections, outlets, as well as DC Volt and ampmeters, temperature control, and plating current-recording equipment, rheostat and master switch, customary in conventional electroforming practices, will be provided by the vendor.

B-1

6. RSD will maintain over-all charge and responsibility for the electro-chemical aspects and for the operation of the equipment. Manpower familiar with the constructional details of the equipment will be supplied by the vendor for the duration of the electroforming of 2 mirrors, but not in excess of 10 days total. This manpower coverage will be on a 24 hr/day basis.

7. Provision is to be included for dismantling the equipment, readying for shipping, at the end of the electroforming process. Crating and shipping charges should not be included.

8. The design engineering will be finalized within 30 working days after issuance of RSD's Purchase Order. Any design engineering necessary after such 30 workday period, which becomes necessary as a result of RSD requested deviations from this Work Statement, can be the subject of further negotiation between RSD and the vendor.

9. Any requirements imposed by RSD on the vendor which materially increase the cost of the contract can be subject to negotiations between RSD and vendor, only if they significantly modify this Work Statement.

PROCESS DESCRIPTION
(FLOW SHEET)

1. Solution Make-Up

Refer to the Flow Diagram for the discussion of the planned operation.

Anhydrous diethyl ether is pumped by pressurized dry nitrogen into the argon-purged solution storage tank. Additions of solids are made from the glove box while a gas-driven stirrer agitates the solution. Cooling coils control the solution temperature during the exothermic reaction of solution. The solution can be recirculated into the storage tank through a filter. A condenser in the vent line minimizes ether volatilization losses, and safety relief valve maintains a pre-selected pressure level in the tank.

The filtration unit operates by alternately decreasing and increasing the pressure in the filter.

2. Start-Up of the Plating Cell

The cathode, after surface pretreatment, and the anode, properly bagged, are placed into the empty plating tank, which is then purged with argon. Plating solution from the storage tank is pumped through the filter(s) into the plating tank. Sparkproof electrical connections are made to the cathode and anode, and plating begins while the plating bath continues circulation through the filter unit.

3. Operating Features

Rapid removal of the plating solution from the plating tank can be made to either the storage tank or outside the plant (i.e. dumped into a deep ditch, river, or large body of water, etc). Return to the storage tank can also be made through the filtration unit.

Removal of the filter units from the process line for cleaning includes a blow-off provision (which should be used to purge the unit while replacing). The properly sized glove box unit above the storage tank will be used for disassembling and re-assembling the filter units. Additions during operation will be made through the solution preparation tank. RSD will supply a current reverser (PR unit) and will connect it to the DC power supply.

Agitation will be provided by the filtering system, which will impinge the solution on the cathode through a hole at the center of the anode.

The following indicative plating parameters will be used:

Anode-cathode	Parallel, at a distance of 2½" both electrodes to remain stationary.
Solution	Aluminum chloride (3M), lithium aluminum hydride (0.4M) in diethyl ether.
Temperature	20-30°C (heating by the plating current)
Filtration	50 gal/hr, continuous
Agitation	Provided by filtration
Cell Voltage	12-16 volts
Cell Current	150 to 200 amps.

Control tests and additions to be provided by RSD.

<u>Item</u>	Solution Preparation and Storage Tank with gas-tight cover
<u>No. Required</u>	One (1)
<u>Purpose</u>	Prepare plating solution within, storage of same, solution make-up to plating tank.
<u>Size</u>	200 gallon capacity.
<u>Geometry</u>	No special geometry required, however, items cited in "Special Considerations" must be taken into account.
<u>Mat'ls of Construction</u>	Glass-lined steel or proven equivalent, as approved by Program Mgr. or Project Engineer. Tank cover sealed with Viton O-ring or gasket designed to "see" vapor only.
<u>Connections Required</u>	1. Bottom drain line (also for quick return). 2. Return line from filter (also solution inlet) 3. Gas purge line 4. Gas vent line
<u>Special Considerations</u>	1. A glove box with air lock should be mounted over tank. Two gloves (neoprene), platform stand to utilize above are required. 12-inch lipped floor around cover is necessary. Height of glove box approximately 4 feet to permit removal of stirrer. Air lock should be no less than 2 feet in any dimension. 2. Vent line should have condenser in series, preferably non-water cooled (Freon, etc). Safety relief valve (2-5 psi) required. 3. Cooling required for tank (preferably not water (Freon, etc). 900 Kcal/hr. heat dissipation required. 4. Gas-driven stirrer, removable or with removable section, to permit closing tank tight (stirrer does <u>not</u> work through cover).

Item	Plating Tank, with gas-tight cover
No. Required	One (1)
Purpose	Electrodeposition Tank
Size	(see geometry)
Geometry	Rectangular tank 40" wide x 24" deep x 50" high
Mat'ls of Construction	Glass lined steel or proven equivalent, as approved by Program Mgr. or Project Engineer. Tank cover sealed with Viton O-ring or gasket designed to "see" vapor only.

Connections Required

1. Bottom drain line
2. Emergency dump line to outside
3. Gas Purge line
4. Gas vent line
5. Filtrate return line

Special Considerations

1. A glove box should be mounted over tank. Two gloves (neoprene) and a platform stand to utilize above are required. A 12-inch lipped floor around cover is necessary. Height of glove box should be 5 ft. to permit removal of electroform and closing tank before opening to atmosphere.

2. Vent line should have condenser in series, preferably non-water cooled (Freon, etc), Safety relief valve (2-5 psi) required in line.

3. Cooling required for tank, preferably non-water (Freon, etc). 2 KW/Hr heat dissipation required.

4. Electrical connections into glove box and into plating tank must be gas-tight.

5. Filtrate return line must be positioned approximately 20 in. from bottom, horizontally centered on the 40" x 50" face and a nipple approximately 1 inch long extent into the plating bath.

Item	Filtration Unit
No. Required	Two (2)
Purpose	Removal of solids, sludge from plating bath.
Size	50 - 75 gallons per hour capacity for each unit.
Geometry	Shape of no concern; cross-sectional area should be suitable to maintain reasonable life.
Mat'ls of Construction	Glass, glass-lined steel or proven equivalent.
Connections Required	(for each unit) 1. Bottom drain line 2. Solution feed line 3. Pressure/vent line
Special Considerations	1. A filter bed of glass cloths and filter aid should be used.

Special Considerations (continued):

2. Provision to remove a unit from the process line for cleaning should be made. Solution blow-out lines which would serve as purge lines on re-insertion should be provided.

3. A three-way solenoid valve, alternately providing pressurized gas and venting to reduce pressure is required. Automation of the process through a current-interrupter based on a timed cycle, a float-activated device, or open and closed contact points is required.

4. Glove box/air lock over storage tank will be utilized for cleaning and reassembling the filters.

Item   Piping, Valving, etc.

No. Required   As necessary for proper function (see Flow Diagram)

Purpose   Fluid transport and control.

Size & Geometry   No special requirements except should be in conformance
                            with sound engineering practices.

Mat'ls of Construction   Glass, glass-lined steel, copper, brass, nickel, teflon,
                            polyethylene or proven equivalent.

Special Considerations   1. For pipe thread connections - no pipe doping compounds -
                            a teflon ribbon should be used.

                            2. Gasketing should be with teflon or polyethylene.
                            (Viton rubber may be used where only contact is vapor).

                            3. Valves should be packless-type. No rubber seal any-
                            where. Teflon with polyethylene a second choice.

                            4. Any sections requiring possible disconnect should
                            be threaded, non-sweat fittings.

                            5. Connections should be kept to minimum.

Item	DC Power Supply
No. Required	One (1)
Purpose	Plating current
Size	Continuous regulation between 10 and 250 amps to 2 -20 volts.
Geometry	Of no concern.
Mat'ls of Construction	Of no concern; electrical leads in contact with solution or vapor should be copper, brass or silver, if insulated, with polyethylene or teflon.
Connections Required	No special connections.
Special Considerations	1. Electrical connections to plating tank through glove box must be gas-tight.

2. Full wave rectification required.

3. Two independently functioning potentiometers to regulate the current density for the plating and the deplating cycle will be required.

4. A mercury-contact heavy-duty relay (good to 250 amps) and preferably operating with 115 volts AC will be necessary. This will be connected to the RSD-supplied current reverser.

STRUCTURAL ANALYSIS OF A PARABOLOIDAL SHELL OF REVOLUTION
SUBJECT TO A GRAVITY LOAD PARALLEL TO ITS AXIS

1.0   INTRODUCTION

The solution to the problem of stress and deformation of a para-
boloidal shell of revolution subjected to a uniform gravity field parallel to
its axis was obtained, based on Novozhilov's "Theory of Thin Shells",
P. Noordhoff Ltd., 1959. As shown in that reference, a pure membrane solu-
tion cannot be obtained for this shell under the given boundary conditions.
The present solution was, therefore, achieved in two steps, i.e. membrane
plus bending solutions.

The membrane solution for the shell does not satisfy the boundary
conditions at the supports. A bending solution was, therefore, added to it,
such that both displacements and slope changes disappear at the boundary. This
corresponds to the case of a thin paraboloidal mirror supported at its outer
circular edge by a ring which is very stiff compared to the mirror.

2.0   OUTLINE OF SOLUTION

The membrane solution was obtained according to the above reference,
pages 107-117, and 155-156. Displacements and slope changes at the supports
were obtained. A bending solution was then derived according to the above re-
ference, pages 288-289. This bending solution contains two arbitrary constants.
These are obtained by stipulating that the sums of the displacements and slope
changes at the supports from the membrane solution and the bending solution
vanish. The determination of the membrane stresses is a statically deter-
minate problem.

3.0   NOTATION
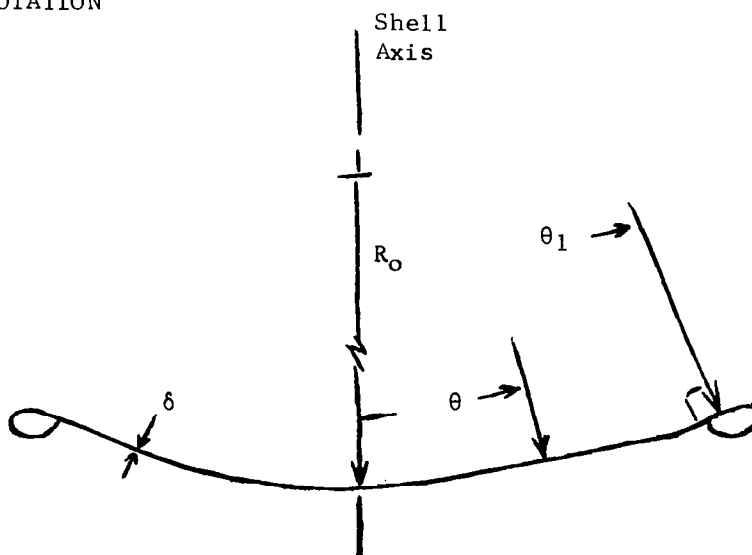
R_o = radius of curvature for $\theta = 0$, i.e. for the vertex of the shell.

ρ = density of shell material

η = number of g's induced by inertia load.

θ = angle between normal to middle surface and the axis of the shell (θ_1 = this angle for edge of shell).

R_1 = radius of curvature of meridian

R_2 = second radius of curvature (= length of the intercept of the normal to the middle surface between this surface and the axis of the shell. (For paraboloids: $R_1 = \dfrac{R_o}{\cos^3\theta}$, $R_2 = \dfrac{R_o}{\cos\theta}$

q = weight of shell per unit area of its surface

$*$ = superscript indicating result due to membrane theory

B = subscript indicating result due to bending theory

T_1 = meridional force per unit length, M_1 = meridional moment per unit length

T_2 = circumferential force per unit length, M_2 = circumferential moment per unit length

 = angle of rotation of tangent to the meridian

δ = shell thickness

E = modulus of elasticity of shell material

 = Poisson's ratio of shell material

σ_1 = meridional stress

σ_2 = circumferential stress

4.0   SUMMARY OF RESULTS

All of the important results of the analysis are listed below. In the first section the results due to membrane theory are given in terms of the physical constants of the shell, and the angle θ, which is defined as the angle between the normal and the axis of the shell. For a vertical axis this angle is the slope of the shell at any point. In the second section, forces and moments due to the bending effect are listed. Actual stresses in the shell are obtained by superposition of the stresses due to membrane and bending effects. In the third section, the maximum stresses and distortion for a shell, in which $R_o = 26$, $\theta_1 = 30°$ and $\delta = .01, .02, .03, .04$ is subject to a 10g inertia load, are shown.

$$w^* = \eta \rho \frac{R_o}{3E} \left\{ \tan\theta \left[\left(1 + 2\gamma\cos^2\theta + \cos^4\theta\right) F_1(\theta) - 3\left(\nu + \cos^2\theta\right)\right] \right.$$

$$- \left[\frac{\cos^3\theta\,\sin\theta}{1+\cos\theta}\right]\left[2 - \frac{\cos\theta}{1+\cos\theta}\right] + \nu\left[2\tan\theta + \frac{\sin\theta\,\cos^2\theta}{(1+\cos\theta)^2}\right]$$

$$\left. - \sin\theta\,\cos\theta\left[F_2(\theta) - \nu\,F_1(\theta)\right]\right\}$$

where

$$F_1(\theta) = \left\{ (\cos\theta)^{-2} + \left(1+\cos\theta\right)^{-1} \right\}$$

$$F_2(\theta) = \left\{ 2 - \cos^2\theta\left(1+\cos\theta\right)^{-1} \right\}$$

$$T_1^* = q\,\frac{R_o}{3}\,F_1(\theta)$$

$$T_2^* = q\,\frac{R_o}{3}\,F_2(\theta)$$

$$q = \eta\rho\delta$$

$$w_B = -\frac{1}{E\delta} \frac{1}{\sqrt{2}} \sqrt{\frac{R_0}{\cos\theta}} c \left\{ (A+B)\cos\beta + (B-A)\sin\beta \right\} e^\beta$$

$$\beta \approx \frac{\sqrt[4]{3(1-\nu^2)}}{3} \frac{\sqrt{R_0}}{\delta^{\frac{1}{2}}} \left[\frac{2\sin\theta}{(\cos\theta)^{3/2}} + \left(\theta + \frac{\theta^3}{12} + \frac{7}{480}\theta^5 \right) \right]_{\theta_1}^{\theta}$$

$$c = \frac{\delta}{\sqrt{12(1-\nu^2)}}$$

$$A = -\left[T_2^* - \nu T_1^* \right]_{\theta_1}$$

$$B = \left[\frac{E\delta^{3/2}}{R_0^{1/2}} \frac{\sqrt{\cos\theta_1}}{\sqrt[4]{3(1-\nu^2)}} \right] \left[w^* \right]_{\theta_1} - A$$

$$T_{1B} = -\frac{1}{\sqrt{2}} \sqrt{\frac{c\cos\theta}{R_0}} \cot\theta \left[(B-A)\cos\beta - (A+B)\sin\beta \right] e^\beta$$

$$T_{2B} = \left[A\cos\beta + B\sin\beta \right] e^\beta$$

$$M_1 = -c \left[B\cos\beta - A\sin\beta \right] e^\beta$$

$$M_2 = \nu M_1$$

4.3   TOTAL STRESSES

$$\sigma_1 = \frac{T_1^* + T_{1B}}{\delta} \pm \frac{6 M_1}{\delta^2}$$

$$\sigma_2 = \frac{T_2^* + T_{2B}}{\delta} \pm \frac{6 M_2}{\delta^2}$$

4.4   COMPUTED RESULTS

Parameters:

R_o = 26.0 inches

θ_1 = 30°

δ = .01, .02, .03, .04 inches

E = 7.98 x 10^6 psi ⎫
⎬ (pure aluminum)
ρ = .1 lb/in^3 ⎭

η = 10

ν = .3